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NOTICES.

In accordance with the provisions of Rule 18 of the Bye-laws, notice is hereby given that those members of Council whose names are placed in italics in the annexed list, will retire from their respective offices at the forthcoming Annual General Meeting.

Mr. Edmund K. Muspratt has been nominated to the office of President, and Dr. W. H. Perkin, F.R.S., has been nominated Vice-President under Rule 11.

Mr. I. Lowthian Bell, F.R.S., Dr. Peter Griess, F.R.S., Mr. John Spiller, and Mr. E. C. C. Stanford have been nominated Vice-Presidents under the provisions of Rule 8; and Sir Bernhard Samuelson, Bart., M.P., and Dr. S. A. Sadler have been nominated ordinary members of Council in the place of Dr. Peter Griess, F.R.S., and Mr. John Spiller, nominated Vice-Presidents under the provisions of Rule 17.

The Treasurer and Foreign Secretary have been nominated for re-election.

Members are hereby invited to nominate fit and proper persons to fill the four vacancies thus created among the ordinary members of Council, in accordance with the provisions of Rule 18. Nomination forms for this purpose can be obtained from the General Secretary upon application.

Members are hereby informed that the Annual General Meeting (1885) will take place in London, on Tuesday, the 7th July, at 11 o'clock, in the Lecture Theatre of the City Guilds of London Central Institute, Exhibition Road, South Kensington, adjoining the International Inventions Exhibition.

The order of business will be:—Report of Council, Election of Office Bearers, and President's Address.

After an adjournment for luncheon, Mr. Frederick Siemens will read a paper—"On heating regenerative gas furnaces by radiation from flame, and on a producer to supply gas specially suitable for that purpose, and for the recovery of bye-products." Discussion on this important subject is invited.

In the evening the members will dine together at the Freemason's Tavern, Great Queen Street. Application for tickets should be made at an early date on the form inserted.

On Wednesday, 8th July, a visit will be paid to Messrs. Joynson & Co.'s Paper Mills, St. Mary's Cray (L.C. & D. Ry.). Messrs. Joynson & Co. will kindly provide luncheon. If possible, and prior to this, arrangements will be made to enable members to inspect Messrs. Orlando Jones & Co.'s Starch Factory, Battersea.

In the evening there will be a Conversazione and Reception by the President of the Chemical Society, Dr. Hugo Müller, F.R.S.; the President of the Institute of Chemistry, Dr. W. Odling, F.R.S.; and the President of the Society of Chemical Industry, Dr. W. H. Perkin, F.R.S., at the Galleries of the Royal Institute of Painters in Water Colours, Piccadilly.

On Thursday, 9th July, visits will be made to the Beckton Gas and Chemical Works of the Gas Light and Coke Company; to the Disinfectant Manufactory, at

A

Barking, and the Pumping Station at Crossness, belonging to the Metropolitan Board of Works; and to the Great Docks now *being constructed at Tilbury*, subject to the consent of the Directors. A commodious river steamer will be engaged for this excursion.

A time-table and detailed programme will be issued with the ballot papers and notices of the number of the Journal for June.

Friday has been left unoccupied to afford members an opportunity to visit the International Inventions Exhibition. Season Tickets, price 10s. 6d., can be obtained from the General Secretary of the Society. Members only are entitled to this privilege.

Members are hereby advised that the Subscription for 1885 falls due at the beginning of the year, and should be paid as soon afterwards as possible. The Council has decided that after January 1st, 1885, the 29th Bye-law will be strictly enforced, so that Members whose subscriptions are more than four months in arrear, will not continue to receive the Society's Journal.

Inquiries having been made as to the transactions of the Newcastle Chemical Society, the Committee of the Newcastle Section have to state that a very few complete sets are remaining, and will be sold to members at £2 10s. per set. A number of separate volumes can also be had by members wishing to complete their sets, at 10s. per volume (generally three years' issue). Application for these should be made to the Local Secretary of the Newcastle Section.

LIST OF MEMBERS ELECTED MAY 23rd, 1885.

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J. H. Brinjes, Whitechapel Engine Works, Fieldgate Street, E., engineer.

A. M. Cheyne, c/o Messrs. Burgoyne, 16, Coleman Street, E.C., chemist.

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Judoo Money Ghose, City College, 13, Mirzapore Street, Calcutta, chemist.

F. M. Haig, Carnforth Lodge, Hammersmith, W., distiller.

T. W. Hand, Public Library, Oldham, librarian.

W. H. Heaton, University College, Nottingham, professor of physics.

J. Wm. James, University College, Cardiff, demonstrator of chemistry.

H. A. B. Leipner, 8, Loftus Street, Sydney, New South Wales, analyst.

Jas. Longshaw, 7, Park Place, Cross Lane, Salford, manufacturing chemist.

T. D. Perkin, Greenford Green, Harrow.

W. H. Richardson, High Field, Southowram, Halifax, science student.

Hugh T. Spencer, Park House, St. Helen's, manager of plate-glass works.

Claude M. Thompson, University College, Cardiff, professor of chemistry.

E. E. H. Thorne, Denia Lodge, West Croydon, chemist.

H. W. T. Wager, 16, Charlotte Street, Portland Place, W., science student.

W. B. Waterfall, c/o Avon Manure Company, Bristol, chemical manure manufacturer.

J. H. Williamson, Golden Hill Works, Stoke-on-Trent, colour manufacturer.

Frank Wilson, The Brewery, Castle Street, Long Acre, W.C., brewer.

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MEETINGS, SESSION 1885—First Monday in each month (unless otherwise indicated).

June 1.—Messrs. Salamon and W. de Vere Mathew—"The *Influence of Phosphates upon Fermenting Worts.*" Mr. H. B. Yardley—"Additional Note on Alumina in Superphosphate." And other papers.

July.—The Annual General Meeting.

Notices of papers and communications for the meetings to be sent to the Local Secretary.

Meeting held May 4, 1885.

COMPOSITION AND MANURIAL VALUE OF FILTER-PRESSED SEWAGE SLUDGE.

(*Paper by* Prof. MUNRO, D.Sc., *read Jan.* 5, 1885.)

DISCUSSION.

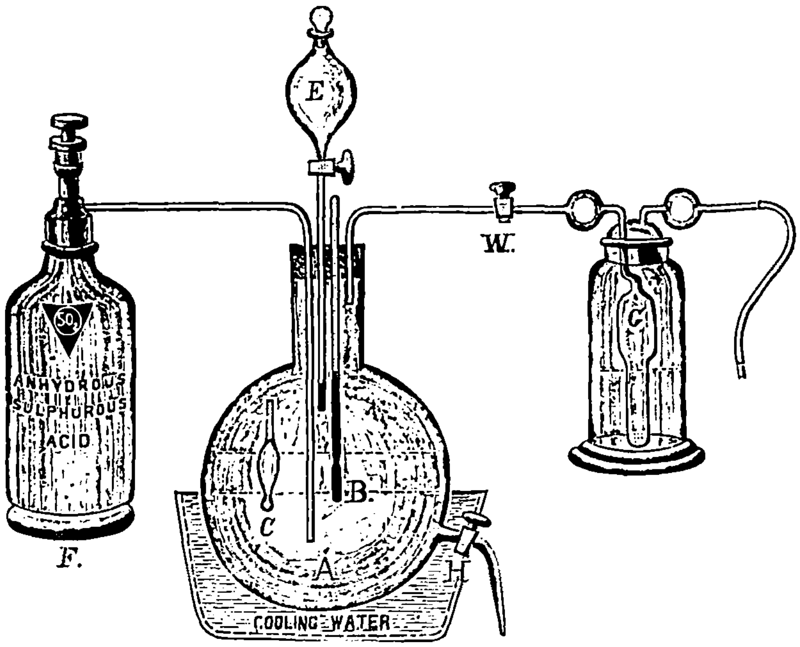
THE manurial value of filter-pressed sewage, as estimated by Professor Munro, was in no sense disputed. The discussion by Messrs. Stanford, Coleman and Tyrer, was chiefly on the condition of the cake when turned out, and on the fact that the manurial value was so low as to preclude its use where much expense had to be added to alter its physical conditions of clogginess and moisture (the latter being put at 50 per cent.), or in transporting the product considerable distances. Messrs. Johnson and Hutchinson contended that it was at least as valuable as farm-yard manure under ordinary conditions, and that the mere mechanical question of comminution was practically accomplished by new modifications of disintegration machines which they were applying.

ON THE PERCENTAGE OF SULPHUROUS ACID IN AQUEOUS SOLUTIONS OF VARIOUS SPECIFIC GRAVITIES, AND ON ITS DETERMINATION THEREIN.

BY WM. B. GILES AND A. SHEARER.

In the course of last year, we had the honour of bringing before the notice of the Society some researches we had made with the view of arriving at a more accurate and speedy method of estimating the amount of sulphurous acid when in combination with bases, and the general result of our work may be briefly summed up as follows :—We found that it was necessary that the sulphite should be added to the iodine solution, and *not* the iodine to the sulphite, and that instead of diluting with water down to the percentage of ·05 per cent. of SO_2, as recommended by Bunsen, it was unadvisable to use any water at all, but simply to place the weighed solid or liquid body straight into an excess of decinormal iodine solution. The results we obtained in this manner were so satisfactory, and so concordant with those obtained by the much more tedious gravimetric process, that we resolved to make a further research as to whether the same method could be applied to the estimation of the acid itself in the free state. A considerable number of preliminary trials having shown us that the determination was as correct in the latter as in the former case, we finally resolved, as a crucial test, to use the pure condensed sulphur dioxide as a means of comparison. The gas (SO_2) employed, was deprived of moisture by passing it through a tube 10ft. long, filled with small flints continually kept wetted with concentrated sulphuric acid, and after being liquefied it was redistilled under pressure in glass vessels, and sealed up in small glass bulbs previously tared without any exposure to the air. In this form the sulphur dioxide appears to be almost chemically pure, the only impurity that could be found being a mere trace of hydrocarbon oil derived from the lubrication of the pump with which its condensation was effected. The glass bulbs, holding from three to four grammes, being weighed, were broken under a slight excess of pure decinormal iodine solution, contained in a stoppered bottle to avoid any possible loss of gas, the amount of excess being then determined as usual with thiosulphate. The mean percentage of SO_2 obtained was 99·68 per cent., and as this entirely confirmed our previous results, we resolved to construct a table showing the amount of this acid in its solutions of varying specific gravity, especially as from various cogent reasons we were led to doubt the accuracy of the only tables we have been able to find as having been made by previous observers. These tables, which are subjoined for the sake of comparison, are three in number—viz., those given by Anthon, Gautier and Wagner, and H. Schiff. As regards the first of these, it appears probable from Anthon's remarks that he prepared his acid by suspending calcic sulphite in water, adding the theoretical amount of sulphuric acid to it, and agitating till decomposition was perfect, after subsidence of the calcium sulphate the supernatant clear fluid was decanted; if this is the case, it is not wonderful that his figures are wide of the truth. With regard to Gautier and Wagner's figures, we have not been able to find what method of estimation was adopted; it may have been a gravimetric one, or Bunsen's mode may have been employed. They agree, however, better with our own results than the others, though there seems to be a constant error throughout them, leading to nearly all the figures for the amount of SO_2 being too low for the respective specific gravities. This might be expected if Bunsen's method was adopted, the free oxygen in the large amount of water, even when long boiled, tending to lower the actual percentage of SO_2. A glance at Schiff's figures will show that they are simply absurd. They appear to be derived from some erroneous theoretical calculations founded—or rather unfounded—upon data given by Bunsen. The results of a very large number of trials, conducted with every precaution that offered itself to our consideration, lead us to propound the following very simple law of the ratio between any given percentage of sulphur dioxide in an aqueous solution at 15·4° C., 760mm. and its specific gravity—viz., that under the foregoing conditions, the percentage of SO_2, multiplied by 5, gives the specific gravity of the solution over and above 1000. Thus, for instance, a solution containing 1 per cent. of SO_2 has a gravity of 1·005, one of 5 per cent. a gravity of 1·025, one of 10 per cent. a density of 1·050, and so on; so that, on the other hand, if we know the specific gravity, we can of course at once tell the percentage of SO_2, provided that this is the only body present to influence the density. Whether this law be *absolutely* correct or not, is, of course, uncertain; but it is undeniable that the experimental results exhibit an extraordinary agreement with the theoretical figures, and that for all *practical* purposes it may be assumed to be so.

The following details of the preparation of the varying specific gravities of the acid solutions, and the volumetric solutions employed, will serve to indicate the precautions that were observed by the authors :—1. The purest re-sublimed iodine obtainable was rubbed to a fine powder with excess of pure potassium iodide, and re-sublimed in the usual manner. It was then fused into a solid cake, and allowed to cool in a closely stoppered vessel. This fusion occasioned considerable loss, but it is the best way of obtaining the iodine in an absolutely dry condition, and the compact fused pieces are less subject to change during the operation of weighing than the micaceous scales which ordinary re-sublimed iodine usually occurs. The titre of the iodine was but slightly altered by this treatment; in fact, the only impurity in the original iodine appeared to be traces of insoluble matter and moisture. Still, when this iodine was titrated in the usual way, it was always found that the weight indicated was slightly less than the actual amount. The cause of this was investigated, and found to consist in the fact that the purest obtainable potassium iodide contains a trace of caustic potass or potassium carbonate, or at any rate destroys a trace of iodine. When this was compensated for, the iodine was found to agree absolutely with sodium thiosulphate, which was twice recrystallised, specially granulated and air-dried till no adherence was observable in the particles of the salt. Two grams of potassium bicarbonate were added to each litre of the thiosulphate solution, as recommended by Mohr, and the solution will, with this addition, keep for months with its titre absolutely unaltered if it be preserved in a cool place and in the dark. If kept in the light it speedily changes. A daily experience, extending over some years of work, enables us to speak confidently as to the unalterability of the iodine solution when pure materials are made use of. Verified burettes, with Erdmann's floats, reading easily to less than $\frac{1}{10}$ of a cc., were employed. The observers worked simultaneously, but with different balances, weights and burettes. Each observation was made at least six times, and the mean taken, in many instances—*i.e.*, in those about the specific gravity of 1040, as many as fourteen to fifteen different trials were made. The specific gravities were all obtained by direct weighings in 100-gramme flasks on Regnault's principle, no hydrometers being trusted to. (The small hydrometer seen inside the flask in the engraving, served merely as an approximate guide to the progress of the saturation.) Particular attention was given to the distilled water employed, as it had long been observed by the authors that an extremely small quantity of a base increases in a remarkable manner the apparent solubility, and hence the percentage of sulphurous acid. As an instance of this, it may be mentioned that if an excess of barium sulphite be suspended in water, the solution, under the influence of a stream of SO_2, rapidly attains a specific gravity of 1060, 15·4° C., yet this solution was found to contain only 0·16grm. of BaO in 100cc., which represents the maximum solubility of barium sulphite in sulphurous acid at 15·4° C. The water used was the middle portion of a distillate from a thirty-gallon copper still heated by steam, and it was boiled for three hours in the absorption vessel itself to get rid, as far as possible, of any dissolved oxygen before passing the stream of SO_2. The same water was used for diluting. The sulphurous dioxide was evolved from the pure liquefied acid contained in glass syphons with regulating screws. The water issuing hot from the still was introduced into the 5-litre flask A, nearly up to the neck. After boiling strongly for nearly three hours, it had decreased till it occupied a space about represented by the dotted lines in the figure, the steam passing away freely from the tap W, which was disconnected from the second flask G. At this period, without stopping the boiling, the sulphur dioxide was turned on from the syphon F, and not till this had saturated the boiling fluid was the lamp removed from under the flask A. The tap W was then connected to the second washer G, and the stream of gas maintained without the least intermission till the desired degree of saturation was attained at the normal temperature. This was reached in about twelve hours, when the lamp was replaced by a vessel of water supplied from the main, which enabled the temperature to be kept constant. This was noted by means of the thermometer B. The small delicate hydrometer C gave the approximate specific gravity. The stream of gas still continuing, the upper part of the vessel A was of course full of gaseous SO_2. To preserve the aqueous acid during the experiments, light purified petroleum oil was now allowed to flow in upon the surface of the acid through the stoppered-tap funnel E to a depth of about half-an-inch, after which the stream of SO_2 was discontinued, and the tap W also closed. By this method of procedure, all access of air from beginning to end of the operation was entirely prevented, and the contents of the vessel could be raised or lowered to any required temperature. A large flask of the same water was used for diluting the strong acid down to the lower gravities, boiled in exactly the same way, and allowed to cool, petroleum being floated on the surface to exclude air. By means of the stopcock H, with a drawn-out point, the acid was then run into the Regnault specific-gravity flasks and weighed, the flasks being tared before each weighing. After weighing, the flasks were then cooled in a freezing mixture, and the acid when sufficiently cooled was transferred to small, very accurately tared and stoppered bottles, which, after re-weighing, were first cooled to about 5° C., in the same freezing mixture, to avoid any loss of gas when the stopper was raised, as it was



invariably found that without this precaution the results were low from loss of gas, and then dropped into a slight excess of decinormal iodine, contained in a stoppered bottle, the excess of iodine remaining being estimated in the usual manner with thiosulphate.

The authors did not attempt to obtain any exact given percentage of sulphurous acid, or to arrive at round numbers in the determinations of the specific gravities, their attention being directed to obtaining correct results with the least possible exposure of the solutions to the air. Finally, they would remark that none of the figures in Table I. are interpolations, but all are results of absolute experiment.

TABLE I.—*Experimental Determination of Percentage of SO_2 in Solutions of various Specific Gravities.*

BY WM. B. GILES AND A. SHEARER.

Observed Specific Gravity.	Temperature.	Observed Percentage of SO_2
1·0051	15·5° C.	0·99
1·0102	,,	2·05
1·0148	,,	2·87
1·0204	,,	4·04
1·0252	,,	4·99
1·0297	,,	5·89
1·0353	,,	7·01
1·0399	,,	8·08
1·0438	,,	8·68
1·0492	,,	9·80
1·0541	,,	10·75
*1·0597	12·5	11·65
*1·0668	11·0	13·09

TABLE II.—*Calculated Specific Gravities compared with Observed Specific Gravities. (The Calculated Specific Gravities obtained by multiplying the observed percentages of SO_2 by 5.)*

Observed Percentage of SO_2	Calculated Specific Gravity.	Observed Specific Gravity.
0·99	1·00495	1·0051
2·05	1·01025	1·0102
2·87	1·01435	1·0148
4·04	1·02020	1·0204
4·99	1·02495	1·0252
5·89	1·02945	1·0297
7·01	1·03505	1·0353
8·08	1·04040	1·0399
8·68	1·04340	1·0438
9·80	1·04900	1·0492
10·75	1·053750	1·0541
*11·65	1·058250	1·0597
*13·09	1·06545	1·0668

TABLE III.—*Calculated Percentage of SO_2 compared with Observed Percentage. (The Percentage calculated by dividing by 5 the units of observed gravity, above 1000.)*

Observed Gravity.	Calculated Percentage.	Observed Percentage.
1·0051	1·02	0·99
1·0102	2·04	2·05
1·0148	2·96	2·87
1·0204	4·08	4·04
1.0252	5·04	4·09
1·0297	5·94	5·89
1·0353	7·06	7·01
1·0399	7·98	8·08
1·0438	8·76	8·68
1·0492	9·84	9·80
1·0541	10·82	10·75
*1·0597	11·94	11·65
*1·0068	13·36	13·09

TABLE IV.—*Tables given by previous Observers.*

ANTHON.

SO_2	Density.
0·95	1·005
1·90	1·009
2·86	1·013
3·82	1·016
4·77	1·020
5·72	1·023
6·68	1·027
7·63	1·031
8·59	1·036
9·54	1·046

GAUTIER AND WAGNER.

Density.	SO_2
1·0028	·5
1·0056	1·0
1·0113	2·0
1·0221	4·0
1·0275	5·0
1·0328	6·0
1·0397	7·0
1·0426	8·0
1·0474	9·0
1·0520	10·0

H. SCHIFF *(Ann. Ch. u. Pharm. 107, pp. 311, 312).*

Density.	Percentage of SO_2
1·0024	1
1·0049	2
1·0075	3
1·0102	4
1·0130	5
1·0158	6
1·0187	7
1·0217	8
1·0247	9
1·0278	10
1·0311	11
1·0343	12
1·0376	13
1·0410	14
1·0445	15
1·0480	16
1·0517	17
1·0553	18
1·0591	19
1·0629	20
1·0667	21

DISCUSSION.

The CHAIRMAN congratulated the authors upon having furnished results which, from their reputation for accuracy and care, could be considered reliable; and the precautions taken to ensure this, especially in the more concentrated, and therefore more volatile solutions, commended themselves to his judgment. He also remarked that as a manufacturer of iodides the authors' observations were well founded, the presence of the traces of free alkali being not accidental, but essential, if stability of this salt was desired.

Mr. TYRER, endorsing these remarks, observed how fully the authors' numbers agreed with those of Mr. Umney, and confirmed by himself when examining the sulphurous acid of the then newly published edition of the *British Pharmacopœia.* These figures were given in the *Pharm. Journal,* March, 1869, as follows :—

Specific Gravity.	Percentage SO_2
1·005	1
1·011	2
1·017	3
1·022	4
1·027	5
1·032	6
1·037	7
1·042	8

Careful precautions were taken to secure uniformity of result, but he (Mr. Tyrer) must bear testimony to the superiority of those used by the authors.

Liverpool Section.

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Vice-Chairman : J. Campbell Brown.

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F. Hurter.
H. Brunner.
A. Norman Tate.
J. Affleck.

Local Sec.: E. G. Ballard, Queen's Park, St. Helens.

Notices of papers and communications for the meetings to be sent to the Local Secretary.

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* It will be observed that these results are not *strictly* speaking comparable at all with the others, the temperatures at which such solutions exist preventing the observations from being made under the standard conditions; in such cases a deviation from the law must be inevitable.

Manchester Section.

Chairman: Sir H. E. Roscoe.

Vice-Chairman: D. B. Howitt.

Committee:

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Wm. Thomson.
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Local Secretary:

J. Carter-Bell, Bankfield, The Cliff, Higher Broughton, Manchester.

Notices of papers and communications for the meetings to be sent to the Local Secretary.

Meeting held April 28, 1885.

SIR H. E. ROSCOE IN THE CHAIR.

THE PRODUCTION AND FIXING OF COLOURING MATTERS BY MEANS OF ELECTRICITY.

BY J. J. HUMMEL.

Since the beginning of this century, when Nicholson and Carlisle discovered the decomposition of water by means of the electric current, electrolytic phenomena have engaged the attention of scientists to a greater or less degree.

In the hands of Davy, as you know, their study led to the analytical production of the alkali metals, and although some attention has been devoted to the electrolysis of organic compounds, that of inorganic substances has generally been the chief attraction.

In 1862, Dr. Letheby, of London, discovered in two cases of poisoning by nitrobenzene that this body was changed in the animal organism into aniline, and this could be distilled from the contents of the intestines and from the tissue of the body.

Letheby sought after the most delicate test for the presence of aniline, and found it in nascent oxygen, which caused mere traces of aniline to give a deep blue colour.

A single drop of a dilute aniline solution, say one gramme aniline in a litre of dilute sulphuric acid ($1H_2SO_4 + 7H_2O$), is placed upon a piece of platinum foil attached to the + electrode of a Bunsen cell. If the liquid is then touched with the - electrode, a deep blue coloured body is gradually deposited on the platinum. Half a milligramme of aniline can by this means be readily detected.

Thirteen years afterwards—viz., in 1875—Coquillon studied the electrolysis of aniline salts.

Coquillon, first of all, repeated Letheby's experiment, and confirmed his observations. After passing an electric current from two Bunsen elements through a concentrated solution of aniline sulphate for twenty-four hours, the + pole became coated with a greenish or bluish-black mass, which could be readily detached. When dry it formed an amorphous black substance possessing a metallic lustre.

Coquillon proved that the presence of a metal, or metallic salt, was not necessary to its production, for he employed carbon electrodes which had been perfectly cleansed by treating with chlorine, nitric acid, etc.

The account of Coquillon's results, in the form of a paper read before the Academy of Sciences in Paris, caused Goppelsroeder to publish in the same year, before the Société Industrielle de Mulhouse, a resumé of his independent work in the same field, with which he had been occupied since the autumn of 1874; and in 1876 appeared a detailed account of the same in his "Études Électrochimiques des Derivés du Benzol," a paper read before the same society.

A pupil of Schoenbein, Goppelsroeder seems to have early interested himself in ozone and its possible industrial applications—*e.g.*, in bleaching, ageing, etc. Eventually, he concentrated his attention on the study of the action of nascent electrolytic ozone on substances capable of yielding colouring matters by oxidation, and, in the first instance, upon a solution of aniline sulphate.

In his numerous researches on electrolytic aniline black, he noticed that the liquid in which the + pole was immersed varied in colour during the experiment, becoming yellow, orange red, and violet, at different periods, and only colourless when the aniline salt was fully decomposed. Filter-paper, cotton, wool, or silk, which was employed to conduct the current from one decomposing cell to the other, became also variously coloured, showing the presence of different colouring matters in the solution. Goppelsroeder showed, indeed, that electrolytic black is a mixture of aniline black with other colouring matters which could be extracted by successive treatment with various solvents, especially water and alcohol. The purified electrolytic aniline black formed a velvety black powder, which, according to analysis, seemed to be the hydrochloride of a base $C_{24}H_{20}N_4$. It may be noted, in passing, that this empirical formula does not agree with that of the aniline black base produced by other methods, which is given as $C_{18}H_{15}N_3$, but it is quite conceivable that there may be blacks of different composition.

From the beginning, Goppelsroeder has continued his researches with great zeal and earnestness, and has indeed made the field practically his own.

He has succeeded in obtaining most interesting results, and a resumé of these I now wish to place before you, illustrated by the series of specimens and elegant album of patterns dyed and printed by the electrolytic method, which Professor Goppelsroeder has kindly lent for the purpose. I am also greatly indebted to him for the liberal manner in which he has supplied me with his various original papers on the subject.

The several chapters of Goppelsroeder's work in which the employment of the electric current is the characteristic feature, are as follows:—

I. The production of colouring matters.
II. The simultaneous formation and fixing of colouring matters upon the different textile fibres.
III. The production of white and coloured discharge patterns upon dyed fabrics.
IV. The production of oxycellulose.
V. The preservation of printing colours liable to be destroyed by oxidation during the printing process.
VI. The reduction of colouring matters for the preparation of so-called vats (indigo, aniline black).

I.

The idea of producing various coal-tar colouring matters by means of electricity originated in the formation of the electrolytic aniline black already alluded to. The electric current was passed through solutions of various salts analogous to aniline hydrochloride, and in each case colouring matter, or a mixture of several colouring matters, was obtained.

It being necessary to prevent the effect of one pole from influencing that of the other, various forms of apparatus were employed, some of which are shown in the diagram.

The solution may be, for example, contained in a simple U-tube, loosely packed with glass wool in the bend, the poles of the battery being inserted in each limb; or the solution may be contained in two separate glass vessels, and the current conducted from one to the other by means of several sheets of filter-paper moistened with the solution; or the solution is placed in the inner and outer cell of an ordinary Bunsen element.

The electrodes employed may be of carbon or platinum; in some cases the negative electrode may be of lead.

The + electrode, which is the principal seat of the production of colouring matter, may be conveniently wrapped in calico. In this case the precipitate formed is deposited between the electrode and the calico, also in the pores of the latter, from which, after washing in water, the colouring matters formed can be removed by means of alcohol or other solvents. In making a qualitative examination of the colouring matters produced, advantage is taken of the fact that if a strip of blotting-paper is partly immersed in a solution containing several colouring matters, the latter rise by virtue of capillary attraction to different heights on the paper. Numerous filter-paper specimens, showing what information may be gained in this manner, are shown in the album exhibited.

The best yield of colouring matter is obtained when the solution surrounding the principal electrode is kept in constant motion.

In most cases the production of colouring matter, or other effect obtained, is to be ascribed mainly, if not entirely, to the decomposition of the solvent employed—viz., water. Oxygen gas is given off at the + pole, and hydrogen at the − pole. The oxygen, either in the nascent state, or as ozone, or by the production of hydrogen dioxide, acts upon the substance in solution, and converts it into colouring matter either by oxidising it or by removing a portion of its hydrogen.

The following is a brief resumé of Goppelsroeder's results relative to the production of colouring matter from various substances under the influence of the electric current:—

1. Aniline hydrochloride in acid and in neutral solution gives emeraldine and aniline black.

2. Aniline salt, with the addition of aniline and ammonia, gives only an insignificant deposit of a brown colouring matter soluble in alcohol, while the liquid acquires a deep red colour, and contains red and violet colouring matters, and these can be obtained by evaporating the solution to dryness and extracting the residue with ether and alcohol.

3. Salts of para-toluidine yield, as the main product, a brown colouring matter; also a little red and yellow colouring matter. Salts of ortho-toluidine yield, on the contrary, a violet colouring matter, similar to that produced by the action of bleaching powder; also small quantities of red, green, and brown and yellow; also emeraldine or aniline black.

4. A solution containing a mixture of 1mol. aniline hydrochloride, and 2mols. paratoluidine hydrochloride, gives a red-coloured solution at the + pole.

By employing a solution of commercial "aniline for red," imperfectly saturated with HCl or H_2SO_4, and with the addition of NH_3, a red and also a violet colouring matter is produced.

It is well known from the researches of Runge, Hofmann, and Perkin, that blue, red, and violet colouring matters are produced by oxidising aniline, or mixtures of aniline and toluidine, and it is not at all improbable that the colouring matters obtained by Goppelsroeder's method may be identical with those obtained by the older methods of oxidation, though I believe this has not yet been proved.

5. Solutions of aniline and toluidine salts, in the presence of potassium-nitrate, -nitrite, and -chlorate, yield at the + pole products from which rose-red and violet-red colouring matters can be extracted, which in their behaviour towards mineral acids remind one of the colouring matters of the safranine group.

6. Salts of methylaniline yield at the + pole a violet, and, under certain conditions, also a blue colouring matter. It will be interesting to know if the violet is identical with the well-known methyl-violet obtained by oxidising dimethylaniline according to Lauth's method.

7. Diphenylamine salts, with or without those of ditoluylamine and phenyltoluylamine, yield at the + pole a blue colouring matter soluble in alcohol. This may, of course, be identical with the diphenylamine blue produced according to the ordinary methods.

8. Salts of methyldiphenylamine give blue and violet colouring matters.

Salts of naphthylamine give a violet colouring matter, accompanied by small quantities of brown, yellow, and red colouring matters.

Turning now to the results obtained from the phenols:—

9. An aqueous acidified solution of phenol itself gives a brown colouring matter, and, under certain conditions, also traces of yellow and orange-red. An ammoniacal solution of phenol gives, under certain conditions, at first a blue, afterwards brown and pink colouring matters, with traces of violet and yellow. With certain additions—*e.g.*, NH_4Cl, KHO, K_2SO_4, KNO_3, the principal colouring matters formed are reddish orange, brownish yellow, and brown and yellow colouring matters, sometimes with traces of violet or red.

10. An acidified solution of naphthol gives as the principal product—a golden yellow colouring matter, possessing a green fluorescence, also a brown and a small quantity of red.

Goppelsroeder has also passed the electric current through solutions containing several substances, partly organic, partly inorganic, in order that by the interchange of certain atoms or atomic groups under the influence of the electric current, new compounds might be obtained.

A solution of magenta containing methyl-alcohol, H_2SO_4 and KI, gave at the positive pole Hofmann's violet.

Very interesting is the electrolytic production of alizarin from anthraquinone.

When the electric current is passed through a boiling solution of caustic potash containing finely-powdered anthraquinone in suspension, a deep red colouration is produced at the pole, due to hydro-anthraquinone, but it is rapidly decolourised under the influence of the air. Only when the caustic potash is extremely concentrated, and the temperature very high, is there a production of alizarin; in this case, during the passage of the current, the melted mass becomes first red (oxyanthraquinonate), then bluish-violet (alizarate), then reddish-violet (mixture of alizarate and purpurate), and then again red (purpurate). If the experiment is continued still longer, the mass becomes brown, then gradually lighter in colour, and finally white (potassium salts of organic acids and carbonate).

The alizarin is obtained by stopping the current whenever the deep bluish-violet colouration is attained, allowing the mass to cool, extracting it with water, and precipitating the alizarin from the solution by adding dilute acid. The yield of alizarin is always small.

At first sight it might be thought that at the high temperature employed, the transformation alluded to would take place without the aid of the electric current, but experiment shows that this is only the case when the heating is continued for a very long time, and even then only traces of alizarin are produced. Further than this, it is noticed that if, when the red colour due to purpurate appears, the current is reversed, the mass becomes again violet, then red, and lastly greyish yellow, since alizarate, oxyanthraquinonate, anthraquinone, and even anthracene are probably reproduced.

II.

As to the simultaneous formation and fixing of colouring matters on textile fibres, the best examples are those of aniline black and canarin yellow.

The following is the arrangement which Goppelsroeder has found suitable for the purpose of printing or writing in aniline black by electrolysis on textile fabrics:—

A lead plate insulated by resting on a sheet of indiarubber serves as the negative electrode. On this rest a dozen folds of calico suitably moistened with dilute sulphuric acid (1 : 10) in order to conduct the electric current, and on these the piece of cloth (silk or cotton) on which the colour is to be produced. The latter is impregnated with a strongly-acidified solution of aniline hydrochloride (about 125grm. per litre).

A second metallic plate, preferably of platinum or gold, serves as the + electrode; this is gently pressed upon the prepared fabric, and the electric current is passed through for one or two minutes. On removing the + electrode, a fixed and permanent impression in aniline black is produced.

If the – electrode is in the form of a stencil plate or an engraved plate, a pattern corresponding to the design upon it is of course produced. Goppelsroeder has also made experiments in printing continuously by means of engraved cylinders.

So rapid, indeed, is the production of the black, that by making the + electrode in the form of a rod of carbon, gold, or platinum, it is possible to write indelibly upon the fabric in aniline black as rapidly as in the ordinary manner.

The characters or design thus produced, after washing with alkaline solution, are perfectly fast, since the fundamental law governing the fixing of colouring matters is properly fulfilled; the colouring principle or chromogen is presented to the fibre in a soluble condition, and is there rendered insoluble.

Goppelsroeder suggests that the electro-chemical method here described might be utilised in print-works, bleach-works, etc., for marking indelibly the piece-ends; also in the customs and telegraph departments.

Dyeing with aniline black may be accomplished by first depositing on the textile fibre a thin metallic layer. Thus rendered a good conductor of electricity, the fibre itself may serve as the + electrode, and be placed in the aniline salts solution; the dyeing would then at once take place on plunging the – electrode into the solution.

Canarin, or perthiocyanogen $C_3N_3S_3H$ is a deep yellow amorphous powder, produced ordinarily by the action of nitric acid or chlorine upon a boiling solution of potassium thiocyanate. It has recently been introduced as a dyestuff for cotton, since it resists the action of light and soap solutions extremely well, and also acts as a mordant for basic coal-tar colouring matters.

By treating the thiocyanates of aluminium and potassium as if for aniline black, and then steaming, "canarin" yellow is at once developed and fixed upon calico; and very probably the remembrance of this process of H. Schmidt caused Lidow and also Goppelsroeder to submit ammonium and platinum thiocyanate to electrolysis.

On passing the electric current through a cold solution of either of these salts, little or no action takes place; but if the solution be heated (preferably to the boiling point), an orange-yellow flocculent precipitate of persulphocyanogen is produced at the + pole, the thiocyanate being entirely decomposed.

Canarin yellow may also be simultaneously produced and fixed upon textile fibres by the method adopted for aniline black. A piece of calico, for example, is impregnated with an aqueous solution of potassium thiocyanate, then spread out upon a number of folds (8-16) of calico, similarly moistened, and resting upon a piece of platinum foil which serves as the – electrode. On placing another piece of platinum acting as the + electrode, upon the prepared calico, and passing the current, the cloth immediately beneath the + electrode is permanently dyed from a canary-yellow to a dark orange, according to the intensity and duration of the current.

III.

Although many colouring matters, as we have seen, are produced by oxidation, most of them are destroyed if submitted to excessive oxidation. This fact is made use of by the calico-printer in the production of so-called discharge patterns—*e.g.*, on Turkey-red and indigo blue grounds. Such discharges may also be readily produced by the electrolytic method.

A piece of Turkey-red or indigo-blue calico is impregnated with a solution of sodium or ammonium chloride or nitrate. Beneath are a number of folds of calico similarly moistened, resting on an insulated sheet of platinum serving as the – electrode. If the positive pole, in the form of a platinum sheet, etc., is pressed on the prepared cloth, there is at once a production of chlorine or HNO_3, according to the salt solution employed, which attacks the colouring matter, colourless oxidation products are formed, and a white discharge on a red or blue ground is the result.

With a view of accelerating the reaction, Goppelsroeder acidified the salt solutions with sulphuric acid. Without such acidifying there was always a production of caustic alkali at the – pole. Albert Scheurer has recently shown that the bleaching action of chlorine gas is considerably more powerful in the presence of caustic alkali, and by substituting caustic soda for sulphuric in his experiments Goppelsroeder also finds the bleaching action to be stronger, and has obtained most excellent discharges, whether the salt used was sodium chloride or nitrate (*Journ. Soc. Chem. Ind.* 478, 1884).

In order to obtain a sharp impression it is necessary to protect the cloth, not covered by the + electrode, by means of glass plates. These prevent the spreading of the liberated gases.

If such salts are chosen whose bases are liberated by electrolysis, and can act as mordants, it may be possible, by dyeing the cloth subsequently, to produce *coloured* discharges. Such might, of course, be at once produced if the liberated oxides were themselves coloured, or became so through being oxidised still further by the electrolytic oxygen.

These ideas thrown out by Goppelsroeder have been partly submitted to the test of experiment, and he finds that by impregnating calico with the usual iron and aluminium mordants, and passing the current through them, as already described, they become mordanted in contact with the + electrode, and produce impressions darker than the surrounding tissue, when subsequently dyed up with alizarin.

He has further attempted, but with what success I know not, what might be called an electrolytic topical style of printing—namely, to produce simultaneously, in presence of the fibre, mordanting oxides, and such colouring matters as would combine with these to form coloured lakes. Under the conditions imposed, this seems an extremely difficult problem.

Black discharge patterns, on red or blue grounds, are readily produced by fixing the electrolytic aniline black on Turkey-red or indigo blue. It would appear that there is not a mere superposition of the black, but a simultaneous destruction or discharging of the red or blue beneath.

Yellow discharge patterns, on blue and red grounds, are produced by means of the electrolysis of potassium thiocyanate; but they seem to be less successful.

A very good yellow pattern, on blue or red ground, however, is obtained by first producing a white discharge, according to the method already described, then impregnating the whole cloth with potassium thiocyanate, and going over the discharged places with the + electrode, when, of course, yellow canarin is produced.

IV.

It is impossible to imagine that in the discharging or production of colouring matter by such a powerful oxidiser as the electrolytic oxygen, the fibre itself should remain unaffected. A few experiments suffice to show that by lengthened action of the electric pen, holes in the fabric are produced, the fibre being destroyed just as it would be by the action of strong bleaching powder.

Before, however, this extreme condition has been attained, Goppelsroeder has proved that in the case of cotton the fibre becomes changed into oxycellulose, just as it is, according to the researches of Witz, under the action of hypochlorous acid.

The most characteristic property of oxycellulose is that it readily attracts and becomes dyed with the basic coal-tar colouring matters without the aid of a mordant, whereas cellulose requires to be prepared with tannic acid to give a similar result.

If a piece of cotton or linen cloth be impregnated with a neutral, acid, or alkaline solution of sodium -chloride, -chlorate, or -nitrate, and be placed upon 8-16 folds of similiarly moistened cloth resting upon a sheet of platinum as the - negative electrode, and if it be then impressed with a platinum plate, serving as the + electrode, after the current has passed through for some time, the products given off at the + pole will so change the fibre immediately in contact with it, that after washing, that portion will dye up in a solution of methylene blue or other basic coal-tar colouring matter, exactly as though it were mordanted with tannic acid. The effect is due to the oxycellulose produced.

On discharging Turkey-red and indigo-blue by the electrolytic method, the cellulose is similarly modified in the discharged portions, so that by dyeing afterwards in various basic coal-tar colouring matters, one may obtain coloured discharge patterns on an indigo-blue ground.

V.

Hitherto, except in the case of the electrolytic production of alizarin, I have referred to experiments in which the positive pole plays the principal part. Let me now refer to other instances in which the negative pole is the most important.

There are several printing colours which, by reason of too rapid oxidation, present difficulties to the calico-printer during the printing process—*e.g.*, the aniline-black printing mixture, reduced indigo, etc. Goppelsroeder proposes to prevent the premature oxidation of such during printing, by putting into the colour-box the – electrode of a small battery or dynamo, and connecting the contents of the colour-box by means of a porous diaphragm, tube, etc., with a smaller supplementary box, filled with the same printing colour, in which the – electrode is placed. The hydrogen given off at the – pole would prevent the oxidation of the colour.

Another service which he thinks might be rendered by the – pole would be that of precipitating, on the textile fibres, the heavy and noble metals, several of which are used in printing. The fabric would require to be impregnated with a thickened solution of the metallic salt, and the - electrode caused to act upon it in order to precipitate and fix the metal.

VI.

The hydrogen of the - pole can also be used for the conversion of the nitro compounds of the aromatic group of bodies into amido compounds. Nitro-benzene can be changed into aniline, for example, and a patent seems already to have been taken out for this purpose; also for the reduction of such colouring matters as aniline black and indigo, to form vats suitable for purposes of dyeing.

In the case of indigo, for example, this dyestuff is finely ground and mixed with a somewhat concentrated solution of caustic alkali. This mixture is placed in a porous earthenware cylinder, resting in a copper vessel, also half-filled with the same alkaline mixture. The copper vessel is connected with the – pole of a galvanic battery; while a strip of platinum attached to the + pole is inserted in the contents of the porous jar. It is well to heat the contents of the copper vessel to the boiling point to accelerate the reduction.

Although hydrogen gas is given off in large quantities at the – pole, in the cold the reduction is very incomplete; but when the alkaline mixture is heated, the evolution of hydrogen ceases, since it then reacts upon the indigo-blue and changes it into indigo-white, which dissolves in the caustic alkali. The characteristic odour of the ordinary indigo-vat is soon perceptible, the solution in the copper vessel acquires a greenish yellow colour, becomes covered with the usual indigo "flurry" or scum, and may serve for dyeing according to the ordinary method.

It is, however, not at all necessary to wait till the indigo is completely reduced, it suffices to allow the current to pass through the mixture for a short time, and then at once alternately to immerse the fibre in the liquid and to expose it to the air.

It is possible even to reduce the indigo upon the fibre itself, so that in the moment of reduction the fibre shall be at once impregnated with a solution of indigo-white, and it requires then only to be exposed to the air to become permanently dyed.

To this end the fabric to be dyed is impregnated with a solution of caustic alkali, mixed with finely-ground indigo, and placed between two metallic plates, one of which forms the +, the other the – electrode. After passing the current through for a short time, and exposing the fabric to the air, it is dyed blue.

With a current of suitable intensity, it might even be possible to dye calico pieces continuously, by passing them (previously impregnated with the mixture of alkali and indigo) through a pair of metal rollers, which formed the + and – electrodes of an electric battery. The cloth would, of course, require to be exposed afterwards to the air and washed.

It is worthy of note that if the passage of the current be too prolonged, the indigo is gradually destroyed, which is quite in harmony with the fact that the indigo is also destroyed in the woad-vat by excessive fermentation.

And now, gentlemen, the first question which is no doubt ready to escape from the lips of many of you is:

Is it possible to apply the electric current in practice in the manner described, and is there any advantage in so doing over the older methods of obtaining the results desired ?

In answer, let me quote from a letter I have recently received from Professor Goppelsroeder. He says :—

"After having opened up this new field of research, as one fact after another comes to view, I become more and more convinced that the seed sown will gradually develop into a plant which will sooner or later bear fruit, and that this new use of electrolysis will some day belong not only to pure but also to applied science. The greater the number of workers in the field the more rapidly and abundantly will fruit be obtained.

"At present I give to science the simple facts just as I find them, without asking whether or not they will find a use to-morrow or only after many years. If in making scientific researches one were always to consider carefully what difficulties might stand in the way of practically applying results obtained, research would often be hindered, if not entirely paralysed.

"In this special electrolytic domain, the question is, whether the new process of producing and fixing colouring matters by electrolysis possesses any advantages over the old method as regards cost and simplicity. Of course the more dynamo machines are improved, and the price of the necessary current is lowered, the more likelihood there will be of the subject passing from the laboratory to the workshop, at first possibly only in a modest degree, but afterwards, perchance, quite universally.

"Should this end ever be attained, there seems to me to be the possibility of electricity being used for the following varied purposes :—

"1. The production of colouring matters and their intermediate products.
"2. The bleaching of textile fabrics.
"3. The dyeing and printing of textile fabrics.
"4. The discharging of dyed colours.
"5. Heating and illumination.
"6. Chemical telegraphy.

"The future alone will show whether electricity is capable of playing such a many-sided rôle."

DISCUSSION.

Mr. LEVINSTEIN : As Professor Hummel has already stated in his interesting paper, none of Goppelsroeder's efforts to produce colouring matters by electrolysis have passed the precincts of his laboratory. It may, therefore, be interesting to mention a patent which has been taken by Ewer and Pick in Berlin for the production of colouring matters containing sulphur (XXII. E. 1258), which, as far as I am aware, has not yet been printed. The method which these chemists propose, combined with the very ingenious apparatus for carrying it into effect on the large scale, appears to me not at all unlikely of being introduced into practice. Ewer and Pick insert two platinum plates or rollers, which form the two poles of an electric current, into a diluted sulphuric acid solution of paramidodimethyl-aniline, containing hydrogen sulphide. As soon as the evolution of hydrogen takes place, the liquor round the anode is coloured blue—this colouration, however, soon disappears, while the anode is covered with a grey precipitate. When this precipitate is removed, the liquor close to the anode again becomes blue, then disappears, and a precipitate is once more formed. This process of colouration of the liquor and formation of colouring matter continues, until at last all the hydrogen sulphide is used up, and the whole liquor becomes dyed an intense blue. The precipitate is the leuco base of the methylene blue. As I already stated, the process appears to me practical, not alone on account of the ingeniously contrived apparatus proposed by the inventors, which you will soon have an opportunity of seeing fully described in our Journal, but also on account of the present unsatisfactory mode of producing methylene blue, by which method useless bye-products are formed, and the yield of valuable blue colour is far short of what theoretically it ought to be. Ewer and Pick claim the avoidance of the production of bye-products, and, as I assume, a quantitative yield of colouring matter

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NOTE ON TITANIC ACID AS A MORDANT.

BY. J. BARNES.

THE property possessed by titanic acid of combining with colouring matters presented itself to my notice during the course of some experiments to discover if alizarin is a reliable reagent to distinguish alumina from other white precipitates which may be thrown down by ammonia—*e.g.*, hydrated silica, phosphates of the alkaline earth metals, etc. I found that precipitated titanic acid gave a reaction very similar to that produced by alumina. This induced me to prepare some cloth with titanic acid, with the view to ascertain whether it would act like alumina as a mordant for colouring matters. The cloth, which had been previously oiled as for Turkey red, was soaked in an aqueous solution of titanium chloride containing some sodium acetate, dried off, and then passed through a bath heated to about 160° F. containing a little sodium phosphate. Prepared pieces of cloth were dyed with various colouring matters, and finally soaped. The colours yielded were as follows :—

Alizarin.—Full red, duller, and of a bluer shade than when alumina acts as the mordant.

Alizarin Orange.—Full orange, considerably redder than with alumina.

Cœruleïn and Alizarin Blue.—Dark green and full blue respectively, very similar to the colours produced on an alumina mordant.

Preparation of Persian Berries.—Brownish orange.

Tannic Acid.—Buff ; very like an iron buff.

Logwood Extract.—Dark grey—almost black.

[*In reply to a question by* Mr. W. THOMSON, *as to the mode of carrying out the alizarin test for alumina,* Mr. BARNES *continued*] : The well-washed precipitate is mixed with water, one drop of a mixture of alizarin and water containing not more than 1 per cent. of pure alizarin is added, and the whole boiled for a short time. The precipitate, if consisting of hydrated alumina, assumes a bright red colour, which is more conspicuous if the precipitate be allowed to subside ; if one drop of weak acetic acid, containing about 1 or 2 per cent. of the pure acid, is then added the colour will not be destroyed. If a red-coloured fluid has been formed owing to the presence of traces of alkali, the addition of the acetic acid immediately destroys the colour. I have sometimes made use of this test to remove any doubt as to whether a precipitate was alumina or hydrated silica. [*In reply to a question by* Mr. SIMS, *as to whether the production of the colour was due merely to the oiling*]: When pieces of the oiled cloth containing no titanic acid were heated with the dye materials, there were practically no colours formed. I have, moreover, obtained similar colours on yarn which had not been submitted to any oiling treatment.

Newcastle Section.

Chairman: J. C. Stevenson, M.P.
Vice-Chairman: B. S. Proctor.
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Alfred Allhusen.
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John Morrison.
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John Pattinson.
H. R. Procter.
W. W. Proctor.
J. F. Stark.
T. W. Stuart.
Local Secretary and Treasurer: J. T. Dunn, 115, Scotswood Road, Newcastle.

Notices of papers and communications for the meetings to be sent to the Local Secretary.

Meeting held April 23, 1885, in the Durham College of Science.

DISCUSSION ON MR. WELDON'S PAPERS READ AT THE LAST MEETING.

Dr. Hewitt: There is one point, which in justice to English industrial chemistry should be stated, and that is in reference to a remark which Mr. Weldon made as to the possibility of making strong bleach from dilute chlorine. He omitted, unintentionally, I am sure, to state that without any mechanical means whatever, that question had been solved in England more than ten years ago. It will be known to every member that about that time Mr. Henry Deacon invented a direct process for the manufacture of chlorine by the decomposition, or, as I should prefer to call it, the combustion of hydrochloric acid with air at a high temperature in presence of a copper salt. That process yields an extremely dilute gas; it has been successfully worked, and it has been clearly established on a manufacturing scale that strong bleach can be made from very dilute chlorine. In order to give some idea of how dilute the chlorine may be, I have myself frequently made bleach testing 36 to 37 with a gas containing 26 or 27 grains of chlorine per cubic foot of inert gases—a degree of dilution far greater than any that M. Péchiney is likely to have used. I think it is due to the memory of Mr. Deacon that this should be stated.

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ON A NEW FORM OF CHLORIMETER.

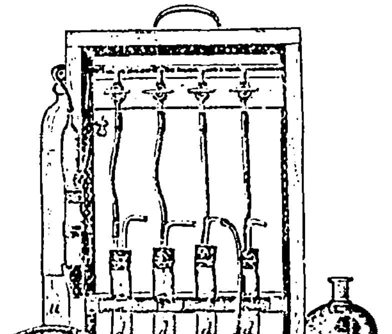
BY J. FLEMING STARK, F.C.S.

Special attention has lately been directed by Her Majesty's Inspector under the Alkali Acts, to the amount of chlorine left unabsorbed in the chambers in the manufacture of bleaching powder. A notice thereon by Mr. Fletcher will be found at p. 30 of his last annual report. Immediately following the publication of that report, a system of testing was instituted at Messrs. Tennant's Works, at Hebburn, which has been continued ever since, and by which, each day, the amount of chlorine remaining unabsorbed in each chamber is ascertained prior to packing. The process first used was as follows:—40cc. $\frac{N}{10}$ arsenite of soda solution was introduced into a bellows aspirator of 0·1 cubic foot capacity; 0·1 cubic foot of gas was then drawn from the bleach chamber, the aspirator well shaken, and the contents then carefully washed out. The amount of arsenite of soda left unacted on was then determined by titration with $\frac{N}{10}$ iodine solution, and the grains chlorine per cubic foot of gas calculated therefrom. A careful examination of this method of testing was made, and its accuracy satisfactorily established.

A short time after this, Ballard's chlorimeter, as exhibited by Mr. Payne at the December meeting of this Section last year, was introduced on the Tyne. This apparatus consisted of a glass jar with attached indiarubber finger-pump, and was a modification of the apparatus described in 1864 by the late Dr. Angus Smith at p. 48 of his first annual report.

The process used was a development of Dr. Angus Smith's "minimetric system or method of analysis." A known quantity of hyposulphite of soda was introduced into the jar along with a little iodide of potassium and starch liquor. Gas from the bleach chamber was then drawn through the solution till a blue colour was obtained. The printed instructions, along with table for calculation of results, are published by Messrs. Mawson & Swan. The results obtained thereby, however, I have found to be considerably under the actual, and this, in view of the possibility of future legislation on the subject, has to be carefully guarded against, as thereby an erroneous idea might be established of the degree of absorption practically obtainable in ordinary working. As regards the reason for the discrepancy referred to, I have not investigated.

Returning then to the bellows test described above, an extended experience proved it to be, though accurate in careful hands, rather cumbrous in working. Each test necessitated a journey from the laboratory to bleach chambers and back again, thus entailing too great an expenditure of time. I accordingly designed the apparatus as shown below, which has now been in daily use for several months. The ease and speed with which it can be worked, and the close degree of accuracy of the results obtained thereby, form my excuse for bringing it under your notice this evening. It consists of a burette *a* for measuring the gas. This is filled with water and connected by indiarubber tubing with the reservoir *b*. Between the burette and reservoir, a tap *c* is inserted, having two holes drilled through the plug at right angles to each other, one of large diameter and the other of small diameter. This admits of either the full flow of water through the tube while the burette is being filled with water, or of a restricted flow while gas is being drawn through the solutions. The four tubes *d*, *d*, *d*, *d*, contain solution of iodide of potassium in water, and can each in turn be brought in connection with the measuring burette by means of the glass taps shown. These absorbing tubes have each two tubes inserted through an indiarubber cork. One tube reaches nearly to the bottom of the solution, and is contracted at its lower end so as to break up the bells



of gas, and at its upper end connected with the interior of the bleach chamber by indiarubber and glass tube *e*. The second tube just goes through the cork and connects with the burette. Between the absorbing tubes and the burette a small wash-bottle *f* is fixed containing solution of iodide of potassium and starch. Between the wash-bottle and burette there is a two-way tap *g*, which admits of the air in the burette being forced out in refilling without having to pass backwards through the wash-bottle.

In using the apparatus, 387·7cc. of gas are drawn through the solution in one of the absorbing tubes. Certainty of the absorption of all the chlorine is obtained by the solution in the wash-bottle remaining colourless. The contents of the tube are then carefully washed out into a beaker and titrated with $\frac{n}{10}$ arsenite of soda solution. The number of cubic centimetres of this solution used multiplied by two gives the grains of chlorine per cubic foot of gas in the chamber.

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ON A PRACTICAL METHOD FOR RECOVERING TIN FROM TIN SCRAP BY ELECTRICITY.

BY JOHN HENRY SMITH, PH.D. (ZÜRICH),

Associate of the Royal College of Science, Dublin.

OWING to the vast accumulation of the above-mentioned material in canister, button, and other manufactories where tinned iron plate is extensively employed, considerable attention has been devoted to the utilisation of the same, chiefly with a view to the recovery of the tin, either in the metallic form or that of one of the various salts of tin used in commerce.

It would be interesting and amusing, although not instructive to an equal degree, to glance at the various methods, patent or otherwise, which have been suggested from time to time to effect this purpose. The separate actions of acids, alkalis, and chlorine gas have been utilised; friction and fusion have been resorted to; and more recently electricity has been pressed into service.

Of these methods I am only aware of three which have been employed on any large scale—viz., dissolving in a mixture of hydrochloric and nitric acids, and subsequent precipitation of tin by metallic zinc; treatment with caustic soda and litharge, forming stannate of soda; and the formation of tetrachloride of tin by the action of dry chlorine gas.

The last-named process is worthy of special mention. It has been described by Prof. Lunge in his report of the chemical section of the Swiss National Exhibition, held in Zürich in 1883.

Some time ago I was requested to recommend some practical method for the utilisation of these tin cuttings, in a district where they could be collected in large quantities. After reading all the literature at my disposal relating to the subject, I was induced to make some experiments, and amongst others, actuated by a knowledge of certain electrolytic processes employed for the precipitation of metals from their ores, I tried the effect of a current of electricity in passing through an acid solution in which a quantity of cuttings were suspended forming the anode, a copper plate serving as cathode. These experiments proved that not only is tin dissolved at the anode, but that it is also deposited in a pure form at the cathode, very soon after the commencement of the action. After continuing these experiments, and making the necessary calculations, I came to the conclusion that this might form the basis of a practical method for the recovery of tin from tin scrap on a large scale.

I recommended that a trial should be made of this process; this was undertaken; the ceremony of obtaining a patent was duly observed, and I was intrusted with the supervision of the erection of the plant, an account of which, and of the results obtained therefrom, I now propose to give you in a brief form.

Although ignorant at the time, I am now aware that at least four English patents have been granted for the same object, effected by very similar means; still, as there are important differences in the process about to be described, and especially as, to the best of my knowledge, it was under my direction that the first plant was erected for the employment of an electrolytic method on any large scale, I venture to hope that the following details may interest the members of our Society:—

General.—The cuttings with which we had to deal varied considerably in value. Some of the thicker ones contained little more than 3 per cent. of tin, while some of the thinner kinds contained 8 or 9 per cent. I considered 5 per cent. as about an average. The quantity obtainable was calculated at about 6 tons per week, and the plant was designed to accommodate that quantity, charging twice a-day. The iron was designed to be converted into sulphate, a large quantity of which could be disposed of at a high price; the remainder was to be converted into "iron mordant"; the tin was to be converted into stannous chloride, and other salts of tin employed as mordants, and largely used by the dyers of that neighbourhood.

Source of Electricity.—No doubt could exist as to the best source for the production of such a strong current as that required, the difficulty was to select the dynamo machine which would be most suitable for the purpose. The machines manufactured by Siemens and Halsk of Berlin had been very highly spoken of as yielding good results in the precipitation of copper on a large scale at Oker in Germany. This firm was appealed to, and one of their small machines was recommended, which, although calculated to perform only about one-half the work required, was deemed sufficient to commence with. This machine is numbered C_{18}, and it is stated to give a current of 240 Ampères with an electromotive force of 15 Volts, and an expenditure of 7 horse-power. (Of course this is considerably higher than the calculated power required.)

Baths.—From the given capacity of the machine it is easy to calculate that the best result would be obtained, without danger of over-heating the machine, by arranging the total resistance to be equal to $\frac{1}{16}$ Ohm. As the subject of the resistance of various liquids to the passage of the electric current has been so imperfectly investigated, and data respecting other factors being likewise unobtainable, it was impossible to determine the best form, number, and arrangement of baths for the purpose in question. The best plan was evidently to make the resistance of each bath as little as possible, consistent with facility of working, and then to arrange them parallel, or in series, or both, so as to include the resistance mentioned above. Eight baths were decided upon. These were made of wood, lined with indiarubber, and each had the following internal dimensions. (I preserve the metrical measurements, not to shirk the trouble of converting them into English equivalents, but because I think every English chemist should be as familiar with the metric system as with our national system of weights and measures.) Length 150cm., breadth 70cm., depth 100cm. The thickness of the wood was 5cm., that of the indiarubber 3 to 4mm. Each four baths were formed by the division of one large tank, 3m. × 1½m., and the two tanks were firmly secured by iron bolts running from side to side. Placed longitudinally they occupied exactly the breadth of the building, and they were fixed at the end of the same, at an elevation of one metre from the ground. In front was a platform, at one side of which was a door for the admission of the cuttings, after being washed and packed. The dynamo was situated in the engine room just behind the baths, and communication was made by two copper cables passing through a hole in the wall. At either side wall, and on a level with the baths, was a dissolving tank capable of accommodating half the cuttings after the removal of the tin. A little farther on, and nearly on a level with the ground, were the evaporating down tanks. The crystallising tanks were situated beneath the ground level, so that the solution could be run off from one stage to another without any pumping arrangement.

Anodes.—These, of course, were composed of the tin scrap. Baskets were obtained to pack the cuttings in. These, at first, were made of wicker-work, but as they were too flexible, and soon rotted by the action of the acid, their place was supplied by strong wooden baskets, whose sides were formed of stout upright wooden bars, 2cm. thick, with spaces between them sufficient to allow the solution to circulate freely, while preventing the exit of the scrap. The internal dimensions of the baskets were:—Length, 120cm.; breadth, 30cm.; depth, 85cm. Great care was required in packing the scrap, because, if it were packed too closely, the metallic surfaces thereby united, and preventing the circulation of the electrolyte between them, necessarily retained their coating of tin. These baskets held from 60 to 70kilos. of the scrap, the 8 being capable of accommodating about half the total quantity required. Long and narrow strips of the same material were employed to complete the communication with the conductor. As the resistance of iron to the current is comparatively great, a large number of these were required to prevent excessive heating. At one end they were soldered together, and connected with the copper conductor by means of binding screws; the other extremities were distributed throughout the scrap.

Cathodes.—Copper plates were employed. These had a thickness of 1½mm., were 120cm. long, and 95cm. in breadth. There were 16 in all, two for each bath, placed one on either side of the baskets. To keep these thin plates of copper as plane as possible, each was surrounded by a framework of copper rod of square section. They were coated with tin to prevent corrosion, as well as to avoid solution by any accidental reversal of the current. These plates rested in grooves at the sides of the tanks, placed at a distance of 10cm. from the sides of the baskets. They were provided, as also were the baskets, with indiarubber rollers extending to the sides of the baths, enabling them to be raised out of the same with ease, and without injury to the indiarubber coating.

Electrolytes.—Dilute sulphuric acid formed the electrolyte. This was employed, not only on account of its comparatively small resistance to the current, but also because it was convenient to turn the solution into the sulphate of iron tanks, as soon as it became saturated with that salt, and all the tin had been precipitated from it. Commercial acid of 60° B. was diluted with 9 volumes of water.

Above the tanks was a pulley arrangement for raising the baskets and plates out of the baths as required; there was also an arrangement of levers and eccentrics constructed, whereby the baskets were kept in gentle motion in the baths, thus exciting circulation in the liquid, and tending to prevent polarisation. The horizontal axis upon which the eccentrics were disposed made about two revolutions per minute, thereby raising the baskets a distance of about 5cm. Levers were fulcrumed into the wall, these passed over the eccentrics, and at their extremities ropes were fixed communicating with the baskets.

The current was conducted by thick copper wires of several plies. Although already coated, they were enclosed in indiarubber tubing as an additional protection.

One of Crompton's indicators, registering up to 250 Ampères, was obtained for measuring the strength of the current, and detecting flaws or breakages in the same. Some such instrument is invaluable for such work, and this proved very reliable.

Results obtained.—The arrangement of the plant having been detailed, it remains to give the results of the working of the same. The critical point had been arrived at; the working capacity of the structure was to be determined; that which calculation could not indicate was about to be demonstrated. Acting in accordance with the principle, "Aspire high," the baths were first arranged in *series*. The connections being complete, the engine of 8 horse-power was set in motion; the dynamo increased rapidly in speed, while the needle of the indicator followed tremulously until it stood vacillating within a few degrees of 240 *Ampères*, the dynamo then making about 900 revolutions per minute. Thus the most economical conditions of working had luckily been hit upon; the machine bore the current bravely, showing no signs of overheating.

Quality of Tin deposited.—This was at the first of a spongy nature, owing to the great acidity of the bath. Soon, however, it began to be precipitated in a more dense, extremely fine, granular, and partially crystalline state, which indeed was preferable, as it fell to the bottom of the bath, and was not in danger of forming a communication with the anode. I will not say that the tin was "chemically pure," but it was purer than commercial tin, and, when thoroughly washed, contained no trace of iron. It fused readily, and almost completely, and that without any addition, provided it had previously been thoroughly washed and dried. The rapidity with which it dissolved in hydrochloric acid was not to be compared to the slow action of that liquid upon granulated tin, and this rendered it peculiarly suitable for the production of stannous chloride.

Quantity of Tin precipitated.—From the data already given, the theoretical amount of tin deposited can readily be calculated. The electro-chemical equivalent of tin as a dyad, compared with silver, is $\frac{117{\cdot}8 \div 2}{107{\cdot}66} = 0{\cdot}546$, and this is equivalent to the precipitation of $67{\cdot}65 \times 0{\cdot}546 = 36{\cdot}94$m.gms. of tin per minute per Ampère. For 240 Ampères, working through 8 baths arranged in series, we obtain a total precipitate of $\frac{36{\cdot}94 \times 240 \times 8 \times 60}{1{,}000{,}000} = 4{\cdot}25$kilos. per hour. As a matter of fact, little more than half this quantity was obtained, the discrepancy being due to part of the current being absorbed in dissolving the iron as well as the tin, as soon as the former began to get bare. This, together with the natural solution of the iron in the acid, led to the rapid accumulation of sulphate of iron in the baths. The acid here employed took about seven weeks to become saturated. On analysis, the baths were found to vary in a very remarkable manner, first one and then another containing the largest quantity of iron. The tin, on the contrary, remained very constant in amount, both in the individual baths and in the total, the average being 1·5grm. per litre. Pure tin was deposited until the acid was saturated and no more tin was present in solution, then hydrate of iron began to form. This might be avoided for a time by the addition of more acid, but it was better to run the liquid into the "green vitriol" tanks, and add fresh solution. It was not at all necessary to continue the action of the current until all the tin had been removed; in fact, after a certain time, the action on the iron was even stronger than that on the tin. It was found in practice that, after the passage of the current for the space of 5 or 6 hours, the quantity of scrap referred to was sufficiently free from tin to be dissolved in the sulphate of iron tanks with the greatest ease; the tin remaining unacted upon in the presence of the large excess of iron always provided for, and it was not difficult to recover that tin, and utilise it with the rest.

Cost of Plant and Expense of Working.—These, of course, would depend upon the neighbourhood fixed upon, and other circumstances; but, in any case, they would be little compared with the value of the tin capable of being recovered. I think I have given sufficient details for any one to be able to calculate approximately their amount for any particular locality. Generally, this scrap is obtained for a mere nominal sum, frequently just for the expense of transport. One stoker and two or three labourers would be quite sufficient to work three tons per week, using one of the small dynamo machines manufactured by Messrs. Siemens Bros. The three cwt. of metallic tin obtained therefrom, at £3 18s. per cwt., will compare favourably with the cost of fuel necessary to maintain the production of 7 or 8 horse-power, wages of workmen, interest on the plant, and occasional repairs. If it were worked on a larger scale, and the tin and iron further worked up into salts, as in the instance detailed, the profits would be increased in a much greater ratio. When we consider that in Paris alone 3000 tons of this scrap are produced annually, it is no unimportant matter to determine the best method of utilising it.

Advantages of this System.—In addition to the consideration of economy, this method possesses advantages which are well worthy of attention, especially where sulphate of iron and "iron mordant" are marketable products. As has been observed, the tin is precipitated in a pure form, and in a state of fine division. It may, therefore, be either fused and sold in the metallic form, or it may be converted into stannous chloride, or other salts, for which it is exceedingly well suited, owing to the ease with which it is dissolved. In the process in which the scrap is acted on by chlorine gas, no choice exists as to the form in which the tin shall be sent to the market, tetrachloride being the constant product. The same remark applies to the method where caustic soda and litharge are employed, and in other respects this latter method has little to recommend it, judging from my own observations. The other process which I mentioned is also unsatisfactory. In employing a mixture of hydrochloric and nitric acids, considerably more iron than tin is dissolved, and when the tin is precipitated by metallic zinc, the contamination of the chloride of iron with zinc chloride renders it of little value. I do not claim perfection for the method I have advocated. It is capable, however, of considerable modification; and I am sorry I have not been able to follow the matter up as I should have liked. A better electrolyte could doubtless be found; probably a solution of stannous sulphate would be the best. I think, however, I have proved that this process is practical, simple, and economical; and further, that it presents the additional advantages of purity of product and variety of utilisation, thus rendering it well worthy of a wider application.

DISCUSSION.

Mr. GATHERAL said: This question of the recovery of tin was one to which a great deal of attention had been devoted, there having been taken out since 1857 no less than about forty English patents on the subject. The question of cost was the thing on which they had chiefly broken down, and he feared this would be the case, too, with Dr. Smith's process if attempted here. In this country the mere cost of collection of tin scrap often amounted to 25s. or 30s. per ton.

Dr. SMITH said that the cost of the tin scrap at the locality spoken of in the paper was only 2 francs per ton.

JAMESON COKING PROCESS.

BY J. JAMESON.

I HAVE been asked to say something to-night as to the coking process associated with my name. I admit that some explanation is required as to its position. A little more than a year ago, and indeed up to a much later time than that, it was considered a highly successful process, and one of immense importance to the coke and coal trade everywhere. At the present time little is heard of it, and that little is sometimes, unhappily, not satisfactory.

In regard to the truthfulness of representations made by the holders of my patents, there is not a word to retract. The results obtained were published far and wide; coals were carried to Felling from nearly all parts of the country and coked in the ordinary oven, usually in the presence of the owner or his representative; the products were carefully weighed and measured; many independent analyses were obtained, samples were sent in all directions, and the fullest inquiry was courted.

When the process was practically adopted, it fell into good hands. Messrs. Bell Brothers converted three ovens, and after some experience with them, seven more; then, with a further experience of many months, another range of seven. The Weardale Iron and Coal Company converted three, then added seven more, and after many months' trial, made a new conversion of twenty, with plant adequate for considerable extension. The results of these trials were reported to us as follows:—That the cost of the process was less than one penny per ton of coal coked; that the yield of coke was increased; that the time required in the oven was shortened; that the quality of the coke was improved as regards sulphur; and our coke, being carefully tried in the blast furnace by itself, was pronounced quite equal to the best Beehive coke. No wonder the use of the process was extended, and that we had very soon installations in a number of different places.

The results, unhappily, were in many cases by no means such as we had anticipated, and complaints began to rise, and, as time went on, to increase. The defects discovered, not of course all at one place, but some at one place and some at another, were the following:—

The yield of ammonia was almost *nil*. The yield of oil was much less than was expected; its quality was depreciated; the suction pipes stopped up; the scrubbers became clogged with a pitchy oil; the passages below the oven floor became filled up with coke dust, or the perforations in the brick bottom through which the gas should pass, closed up; the gas became unburnable or explosive. The Directors of the Company found themselves embarked upon a sea of troubles with no autocrat to direct, and no absolutely certain programme to follow.

I am, I confess, greatly tempted here to indulge in some personal explanations ; but they would have little general interest, if any, and I refrain.

The Directors of the Company did not cease to believe in the process. Most excellent results had been obtained, and the difficulties arising appeared to be mechanical and not inherent in the invention itself. The falling off in results for the reason that it was due in one place to one cause, and in another place to another cause, was not unhopeful ; and without dividing a penny, the Directors (being at the same time the largest shareholders) have been content to spend, in the prosecution of the enterprise of the Company, upwards of five thousand pounds, independently of the cost of all preliminary experiments.

Now, as to the present position of affairs. I do not propose to take you through all the tedious steps of our discoveries. Alterations in ovens or apparatus involve the expenditure of considerable time, and often a much longer time is required for practical demonstrations of their effect ; but, perhaps, the longest time of all is occupied in localising the exact cause of failure where there may be several causes.

The first and great cause of all our trouble, after many experiments, was conclusively proved to be ingress of air at the oven bottom, and the first requisite to success, therefore, is that the oven bottom be air-tight, or as nearly so as possible. This point was strongly pressed in all our circulars and in all our directions for installation, but, as it proves, not sufficiently so. Any ordinary building of bricks and fireclay or (unless carried to both a great depth and width) of bricks, fireclay, and tar, becomes after use porous, and admits air. The effect of leakage of air is extremely bad in many ways. The air is heated in its passage through the hot brickwork, and meeting with the warm gas from the oven (dependent upon degree of admixture and temperature) it decomposes the ammonia, and the oil or it makes an explosive gas ; or the air may burn in the gas atmosphere, just as gas does in air—it produces a heat in the pipes which is very undesirable, causing a distillation of the oil deposited on the sides of the pipe, with the result that the lighter fractions of the oil are carried forward with the gas, and the heavier part is formed into pitch on the pipe, and gradually stops it up. There is a further injury caused by the ingress of air —namely, that all useless air tends to carry away ammonia and oil, by hurrying the gas dealt with through the scrubber and condenser. If the gas be mixed, for instance, with its own bulk of air at equal temperature, it is clear that the condensing apparatus must be double the size, and that scrubbers and exhausters possess only half the efficiency they would have if the air were excluded. A simple method of discovering the tightness of an oven bottom is to cover the whole of the perforated floor with papers wet with oil, and then to apply considerable suction, and estimate the quantity of air passing. But, as defects may arise in this respect at any time, it is also of great importance to have a simple means of ascertaining how much gas is passing from every oven to the main when in regular work ; and to accomplish this we put a regulator valve in the pipe of each oven between the oven and the main. This regulator is a valve so marked as to indicate exactly the amount of opening through it, and there is a syphon water-gauge coupled to the pipe at one end on one side of the regulator, and at the other end to the pipe on the other side of the regulator, so that the indicated difference of level shall show the exact pressure tending to pass gas through the restricted opening. Within very narrow limits, this enables us to estimate the exact quantity of gas passing from each oven.

If from a 5-ton oven, burning its charge in 72 hours, there is a passage of gas of about 1000 feet per hour, with a good suction on the oven, it may be presumed that the process is working well ; but if the passage of gas be much more or less than this it points to some defects which should be investigated and remedied.

Next to the tightness of the oven bottom, I think I should attach importance to the construction of the oven floor ; and the best floor, in my judgment, is one formed of alternate firebricks and T irons ; the bricks being shaped so as to take in the T iron, and form a level floor. I show a piece of T iron which has been in an oven continuously worked for four months. It is practically as good as new.

I show a drawing of the special brick and of the oven floor with T irons in place. To provide for expansion and prevent the T iron from rising into the charge, we cut these into pieces not more than 18 inches long, and bevel the ends as shown in the drawing, so as, while affording room for expansion, to prevent actual opening for the passage of coal into the channels below ; or the floor may be constructed in accordance with plan No. 2. The T irons prevent an action which has caused a great deal of trouble —namely, when the openings through or between the bricks enlarge downwards, as they must do to prevent stopping up, any chipping of the top makes an opening equal to the size of the enlargement at the depth to which the chip extends. The heavy rakes in use for drawing coke chip the bricks, the openings become much too large, and coal and coke dust get down and impede or stop up the passages below. The T iron, however, will bear a heavy blow of the rake and distribute it on a large solid body of brickwork so as to do no harm to it.

Next in importance to the points already referred to, I attach value to an improvement effected by a slight cooling of the gas of each oven separately before passing into the main.

It is very easy to reduce the temperature of gas at 400° to 150°, a very short pipe will do it ; but if gas at 400° be mixed with gas at, say, 150°, so as to make the resultant temperature 160°, it requires a great surface of pipe to extract the 10° thus added and bring the temperature to 150°. There is, however, a still stronger reason for the partial cooling of the gas of each oven separately—namely, that gas and products of different quality come off at different periods of the process, and if hot gas from an exhausted or nearly exhausted oven be mixed with rich gas from a half-burned oven, the products in the latter may be decomposed ; the oil, which from this half-burned oven is condensible, may be so acted upon that part of it is converted into permanent gas, the remainder is injured in quality, and the ammonia may be wholly lost. With cooled gas, however, this action is stopped.

I show a drawing of the arrangement of the ovens showing the cooling pipes, details of the regulator valve, the suction pipe of each oven, and its cleaning arrangements.

I think it is well to condense the steam used for the steam jet, so as both to recover the oil and ammonia brought down by the steam, and at the same time to make the gas available for burning ; but, of course, if a mechanical exhauster be used this is not necessary.

The modifications I have referred to have been practically applied at one installation, as follows. Formerly half-an-inch suction on the oven bottom overheated the pipe to the extent of sometimes making it red-hot, and as the chimney draught was a quarter-inch, the real pull on the gas was necessarily exceedingly small. With half-an-inch of suction the quantity of gas and air passing from the oven was equal to 80,000 feet per ton of coal coked, by far the larger portion being, of course, air. Now, with the same oven, 3½ inches suction gives a passage of gas equal to less than 12,000 feet per ton. The temperature in the oven pipe with this great suction is usually under 200°, and rises only to 400° as the oven gets nearly burned off. The yield of oil is increased to four times what it was, and, in fact, both in ammonia and oil the Felling results have been obtained. The scrubbers, cooling pipe, and steam jet, are equal to more than six times the work they could formerly do; they can now operate six ovens instead of one with equal efficiency, and the quality of the oil is greatly improved. There is no possibility of burning the oil by reason of defect in one oven of the range; and any defect in stoppage of floor or leakage of air is at once apparent. The attention required is diminished, and I confidently believe that the application of the remedies devised will meet our troubles at every installation. That is to say, there is not any defect which has arisen, at any installation, which is not, so far as I know, met and covered by the modifications introduced. Experiments are still going on with a view to discover the best and cheapest means of making a really air-tight bottom, but as we can safely go to a depth at which the temperature never rises above a dull red, there cannot possibly be any permanent difficulty about it.

DISCUSSION.

Mr. JOHN PATTINSON was interested to know that the modifications spoken of were making the process a working success: he asked whether the sulphate of ammonium was recovered at any installation, and to what purposes the oil was best applied. Mr. Jameson was also asked the strength of the liquors yielded.

In reply, Mr. JAMESON said: The ammonia is not worked up into sulphate at any of our present installations. The largest number of converted ovens at one place is 54, coking about 600 tons of coal per week; these ovens are not yet modified in accordance with the arrangements described in the paper; the coal used is one yielding only small results, and the yield of ammonia is not large enough to warrant the erection of sulphate plant. The quantity of ammonia yielded depended on the coal, and varied from 2 or 3lb. to about 15lb. (expressed as sulphate) per ton. As to the oil, Dr. Armstrong, of London, is going to read a paper at the Iron and Steel Institute meeting shortly, which will I believe deal with the whole question of its nature and uses. Dr. Armstrong told me that he finds it in a very marked degree a low temperature product, and hence especially adapted for conversion into other forms by higher temperature. He suggested its application as a source of illuminating gas, and that fractions not suited for other purposes should be used as a liquid fuel for steam-ships. A good deal has been used for creosoting and found very good for the purpose. The strength of the ammonia liquor varied from 2 to 5oz. per gallon.

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THE ST. BEDE PATENT DECOMPOSING FURNACE.

BY W. L. RENNOLDSON.

THIS furnace, which is the invention of Mr. Thos. Larkin of East Jarrow, has been designed for the manufacture of sulphate of soda, hydrochloric acid, and other substances. In its arrangement every attention has been given to the important requirements of the present time, which are, capability to make salt-cake of first-rate quality, and to yield the HCl in the largest possible quantity, and this of high density (so as to allow of the best results being got in the bleach department), while at the same time to admit of the escaping flue-gases being readily maintained within the standard limit of acidity.

These desiderata are all accomplished with this furnace, and not only accomplished, but with remarkable economy under every item—viz., labour, fuel, vitriol, first cost and maintenance. In its general character the furnace is not such a radical departure from the hand-worked furnace as other mechanical decomposers are, the aim having been to retain the really efficient features of the best hand system, and combine with these the several improvements which constitute the invention, and which were actually desirable.

The plant consists, in the first place, of an ordinary pan situated so that the charge of salt is dropped in from a wagon on an overhead tramway, and it (the pan) is fired by the waste heat from the roaster.

The roaster is of the close or muffle style, but with a specially low and perfectly flat inner roof (or arch), consisting of sections of iron plates supported in a very novel manner, and the space between it and the upper arch, which is also nearly flat, is divided into independent transverse compartments or channels.

The heating of this roaster is effected by a double series of small fires; one series heats the bed from its under side, the other heats the roof. Each fire has its own flue across the furnace, and afterwards all these small flues lead into a main one, from which all or any portion of the waste heat can be directed under the pan.

The mechanical appliances for working the batch in the roaster, are a vertical shaft, and horizontal radial arms with scrapers, and being very strong—*i.e.*, strong in the proper place, but by no means ponderous—are very serviceable and efficient.

A small steam engine transmits the power from below, and the whole arrangement works smoothly and silently.

The substitution of steam-power for manual labour for working the batch in the close roaster, and the protection of every part of the steam-worked tools from the direct action of the fire, are special features of the invention. And the adoption of several small fires in place of one enormous, and more or less unmanageable fire, is an improvement of the greatest advantage.

The multiplication of the sources of heat leads to an even heating of the whole furnace, and, as the coaling and cleaning (scarring) is done at intervals, and at one fire at a time, the temperature of the whole furnace can be maintained with great regularity, and thus the great cause of destruction to the structure—*i.e.*, expansion and contraction—is largely reduced. And as each flue, under the bed and over the arch, has its own fire, and each flue has a damper, it becomes an easy matter to manipulate the heat in any way, or even to entirely lay off any section of the arch, in the event of a failure occurring.

These flues being on the "flash" system (in contradistinction to the usual "wheel" or return system) a very slight draught to the chimney suffices, so that any leakage would not be of acid gas to the chimney, but of smoke to the condenser, owing to the (with this furnace) *undoubted* stronger draught to the condenser than to the chimney; however, the useful provision above mentioned effectually provides against leakage in either direction.

The fact that all the stirring and raking of the batch and the evolution of its HCl takes place with *closed working doors*, is a very great advantage in many ways—direct and indirect, more especially when coupled with the fact that the roaster is a close one.

There is the highly important one that it renders it a matter of ease to obtain practically all the hydrochloric acid in a highly concentrated state without any costly ranges of cooling pipes, or extravagant condensing space; this is a considerable saving in cost for plant and its maintenance, and the cost for *wash towers* and for their supply of water are entirely saved. The high strength hydrochloric acid means a less volume of itself, and of the still liquors, and as the loss of HCl, as free acid, in the still liquors is in direct proportion to their volume, there is a greater utilisation of the acid, and consequently a larger production of bleach per ton of salt decomposed.

From the same cause there is a saving of chalk or limestone for neutralising, while less still, etc., room and steam suffice for working such liquors.

Another advantage connected with the closed working doors is in the matter of the usual escapes of acid vapours about the furnaces—*i.e.*, the "*low level escapes*"—which are the cause of much anxiety in many neighbourhoods; with this furnace such escapes are almost unknown.

This satisfactory state exists partly from the doors being closed, and also from the reason that a much stronger draught to the condensers is used with these furnaces than with hand-worked ones.

A strong draught *on hand roasters* is the cause of several evils; when the working doors are open, as they must be to a large extent, the inrush of air rapidly cools the roaster, and heats the condenser, and so interferes with efficient condensation.

Then, again, the practicability of working with closed doors and strong draught prevents the possibility of any acid gas leaking into the chimney gases through any crevice that might exist in the arch, etc.

This arrangement of furnace also benefits the workmen to a very considerable extent. The drierman is relieved of nearly all the punishing nature of his work. The steam-worked tools relieve him of the hard labour, heat and gas he had to bear with the hand furnaces, and the closed doors and strong draught keep the air about the place free from acid; and the attending to the several small fires does not give him the severe scorching that the ordinary large fire does; and when he draws the batch, it is so well worked and evenly fired that not even then is he troubled by gas.

Even the pan-man is much benefited, as he is allowed to have a stronger draught than formerly, when there was always a fear of drawing dangerous heat, etc., from the roaster through his open, or perhaps leaky partition damper. He is also relieved from the scorching which the ordinary pan-fire gave him.

The sulphate can readily be made of any strength and with great uniformity, and this uniformity of composition and of physical condition, being free from hard lumps and fine without being dusty, renders it highly suitable for the manufacture of alkali and of glass.

The following is a statement of results :—

RESULTS OF WORKING BY THE ST. BEDE CHEMICAL COMPANY, NEWCASTLE—AVERAGES OVER LAST THREE MONTHS.

(Three furnaces are now in constant operation.)

Salt decomposed per furnace per week (six days) (80 tons have been done in a week of six days).........	72 tons
Sulphuric acid consumed (on weight of damp salt as received)...	78 per cent.
Sulphate (salt cake) made	110 per cent.
Test of sulphate, 0·4 acid	1·3 per cent. salt
Test of gas from pan and roaster at the condenser inlet	175 F.
Test of gas from pan and roaster at the condenser outlet	0·19gn. HCl per cubic foot
Test of gas, etc., in chimney...	0·07gn. HCl per cubic foot
Test of hydrochloric acid produced	29° T. at 60 F.
Bleaching powder, 1 ton obtained from each...	46cwt. salt

	Per Ton Sulphate s.	d.
Labour (all at and about furnaces)	2	0
Labour since reduced, now working at	1	9
Steam, fuel and labour for..................................	0	1¼
Fuel at pan and roaster, 7½cwt. small coal	1	3

Repairs.—During the fourteen months the system has been at work there have been no appreciable repairs, no renewal of arms, nor of bridge, arch, or casing of either pan or drier fires, only replacing of worn scrapers.

DISCUSSION.

Dr. Hewitt said that he had seen the furnace at work that morning, and observed that both the method of firing and construction of the arch or roof were novel. He considered that the idea of heating with a number of small fires with separate flues was a great improvement on the one large fire system, because the latter caused great intensity of heat in a part only of the furnace, with consequent unequal expansion and contraction. He received with reserve the statements as to cost of maintenance, owing to the short time during which the furnace had been at work; but at the same time he thought that its special construction was likely to conduce to economy in that direction. As to salt cake, he preferred its being made in two operations, as in the pan and roaster of the hand-worked furnace. But in this, mixing had to be done by hand, and the quality of salt cake produced depended on the skill of the workman. Moreover, hand mixing necessitated open doors, leading to both influx of air and escape of gas. Open mechanical furnaces, intended to replace the old "pan," had never seemed quite satisfactory to him, owing to the dilution of the gas with air. Gaskell and Deacon's, and Gamble's plus-pressure furnaces had been intended to meet the objections to the second part of the operation, inasmuch as they were "close," and permitted little, if any, dilution of the evolved hydrochloric acid. But Mr. Larkin had added to the old pan a mechanical muffle, in which thorough incorporation was effected by a mechanical stirrer with closed doors, thus effecting better condensation, and avoiding low-level nuisance. The stirrer is not exposed to a very high temperature, and its wear and tear is reduced to a minimum. Altogether, both as regards fuel, condensation, and make of bleach, he thought very highly of this furnace. Having regard to present circumstances, which rendered it desirable to make a maximum of bleach with a minimum of soda, and to obtain a more condensed, and thereby stronger gas, there was no doubt that Mr. Larkin's furnace was a step in the right direction.

Mr. Stuart: It would have added materially to the value of Mr. Rennoldson's interesting paper, had he mentioned the particular form of decomposing furnace over which he claims advantages for the Black and Larkin furnace, and I think it would have been well had he selected for comparison the furnace which in his opinion best suits the requirements of the present day. All other things being equal, as to cost of plant and sulphate, combined with satisfactory quality of the product, that furnace is the best, in the present condition of the Leblanc process, which produces a ton of bleaching powder from the least quantity of salt. On the one hand we have the Jones and the Mactear mechanical decomposing furnaces, and on the other the Deacon and the Gamble plus-pressure hand furnaces. In discussing the merits of the Black and Larkin furnace to-night, it will be obviously necessary to select for comparison from these four, the furnace which is best adapted to our present requirements. The Jones and Mactear furnaces have each proved a mechanical success, and the sulphate produced by them is of excellent quality. Per ton of salt decomposed, however, the cost for these furnaces is enormous, and in connection with the now all-important question of salt consumption, per ton of bleaching powder, represented by the condensation, I venture to express the opinion that these furnaces have been superseded. Being open furnaces, the principle is wrong, the hydrochloric acid being highly heated and diluted with the products of combustion and excess air, and consequently is in that condition in which it is impossible to obtain satisfactory condensation. Heat and dilution constitute the difficulties in condensing hydrochloric acid, and of the two, dilution is the more prejudicial. The Jones furnace and the Mactear furnace have been superseded by the plus-pressure hand furnaces, which have proved so satisfactory that they have been largely adopted by alkali manufacturers. Being close and not open furnaces, the hydrochloric acid is evolved at a very low temperature, and not mixed with the products of combustion, which go direct to the chimney. The only dilution of the hydrochloric acid arises from the small amount of air that enters the doors of the roaster, when the men are working and drawing their batches. Instead, therefore, of the hydrochloric acid being evolved highly heated and diluted, as in the Jones and Mactear furnaces, it is in the plus-pressure furnaces produced at a comparatively low temperature, and being only slightly diluted, is easily condensed into acid of high density for the stills. Comparing the Black and Larkin furnace, which I have carefully examined several times, and the plus-pressure hand furnace, it will be seen, as specially pointed out by Mr. Rennoldson, that the sulphate is roasted with closed doors, air being only admitted for a short time when the batches are being drawn, and consequently there is less dilution of the hydrochloric acid in the Black and Larkin than in the plus-pressure hand furnace. Then, the Black and Larkin furnace is also worked with a plus pressure, got by the very simple means explained by Mr. Rennoldson. Excellent, therefore, as the condensation is in the plus-pressure hand furnace, it must be better in the Black and Larkin, in which also there is complete removal of the low-level gas, and the obvious advantages of mechanically worked sulphate as against hand labour. Compared with the plus-pressure hand furnace, I do not think there is any saving on plant or fuel. Wages amount to 2s. in the Black and Larkin against about 2s. 10d. in the plus-pressure hand furnace, but this will be partly at least covered by the extra repairs on the machinery. Mr. Rennoldson states that the consumption of O. V. with the Black and Larkin furnace is only 78% on salt, and although this is a very low figure, I am inclined to think that it must be rather less than in the plus-pressure hand furnaces. I think it would be difficult to show a direct financial saving in favour of the Black and Larkin over the plus-pressure hand furnace, which is really a very satisfactory one, but the well-recognised advantage of a really efficient machine over hand labour, the complete removal of the low-level nuisance, better condensation, combined with the durability of furnaces, which I have quite satisfied myself about, constitute the advantages which the Black and Larkin possesses over the plus-pressure hand furnaces, and which justify me in expressing the opinion that it is the furnace by which bleaching powder can be made from the least consumption of salt, and also that it is the furnace, taken as a whole, which best suits the requirements of the present day.

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ON SECURITY OF DOCUMENTS.

BY J. JAMESON.

This paper described the importance of mordant in writing ink, and the dangers which would arise from the use of ink containing no mordant, such as a mixture of lamp black and gum water, and it then described means of protection against the use of such ink. It referred to dangers arising from the use of a film on postage stamps soluble in water, but impervious to obliterating ink, which rendered the present system of obliteration inefficient, and finally dealt with a new application of soluble films on paper for transformation advertisements, Christmas and birthday cards, etc., the arrangement being such as that washing with water removes all printed matter, which is carried on the filmed surface, leaving any printing below it intact. Some very striking examples of transformation were shown, and samples were handed to the members. The protected paper and transformation pictures are shown at the Inventions Exhibition, the latter as Reid's patented system.

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Meeting in the Rooms, 207, Bath Street, on Tuesday, May 5.

MR. G. BEILBY IN THE CHAIR.

WOOD NAPHTHA.

BY MR. C. A. FAWSITT.

As the distillation of wood and the working up of the resulting products form an important branch of the chemical industry in the West of Scotland, I thought it perhaps might be of interest to some of the members of this Society, if I read a short paper on what, in this district, is termed "wood naphtha," but elsewhere generally "wood spirit."

In the first place I shall say a few words on the composition of wood. Secondly, give some conditions on which the yield of the different products of wood distillation depends. Thirdly, dwell more particularly on the preparation, properties and uses of wood naphtha.

1st. As to the composition of wood.

Wood consists principally of cellulose, impregnated with incrusting material, such as resin, volatile oils, sugar, gum, tannin and protein bodies, besides the usual mineral constituents of plants.

The amount of incrusting material varies, being more abundant in hard and heavy varieties than in light and soft kinds; and wood which contains it in the largest proportion gives the most acid and naphtha on distillation. These incrusting materials contain more carbon than cellulose, and the greater the amount of carbon and hydrogen in wood compared with cellulose depends on a greater amount of these bodies. Beech wood was found, by Payen, to contain 40 per cent. cellulose and 60 per cent. incrusting material.

Wood is a highly hygroscopic substance, as is shown by the fact that if placed in air of ordinary temperatures, after having been dried completely at about 105° C., it takes up about 20 per cent. of water. Its average composition is as follows:—

	Carbon.	Hydrogen.	Oxygen.	Nitrogen	Ash.	Water.
Dried at 120° C.	50%	6·0	42·0	1·0	1·0	——
Air-dried	39·2	4·7	31·5	0·8	0·8	20·0

2nd. Some conditions on which the yield of different products of wood distillation depends.

When ordinary air-dried hard wood is distilled in closed vessels, there results

Charcoal,	which amounts to	21 to 26%	of weight	of wood.
Liquid distillate	,,	50 to 55	,,	,,
Permanent gas	,,	20 to 25	,,	,,

You can readily understand that the weight of the different crude products depends on a variety of conditions.

The first I will mention are the species, age and condition of the wood, and also the soil it is grown in.

As regards the species, it may be said that hard woods are preferred by the wood distiller; and of these oak, beech and birch are most used in this district. The following example shows the difference in yield of acid for 100 parts oak and Scotch fir:—

	Crude Acid.	Acetic Acid.	Tar.	Charcoal.
Oak	47·6	5·1	6·1	21·9
Scotch Fir	45·0	2·7	10·0	28·0

You will notice that, although the weight of crude acid in both cases is much the same, the amount of acetic acid is very much less in the case of the Scotch fir; and I should like to mention here that the yield of acetic acid and naphtha always varies in the same direction—*i.e.*, if the yield of acid is large, so is that of the naphtha, and *vice versa.*

The condition of the wood when distilled has an important influence, sound wood giving much better results than unsound. It should also, previous to distillation, have lain for some time, in order that a considerable proportion of the water might escape. This is then called "air-dried" wood. It appears to lose water for about 1½ to 2 years after it is cut, and should not be used, at the soonest, within one year of the time of cutting.

New beech wood contains....................30 to 35% H_2O
,, ,, well air-dried, contains 16 to 20% H_2O
Trunk wood gives more acid than branch wood.

We will now notice another important set of conditions—namely, temperature, size and shape of retorts.

The temperature has a most important influence, and it can be taken as an invariable rule that a low heat gives more charcoal, acid, naphtha and tar, but less permanent gas, than a high one; and this can be readily understood if we remember that at a low heat bodies are formed, rich in oxygen, such as $C_2H_4O_2$ and CH_4O; but at high temperatures these same bodies are decomposed into compounds rich in carbon and hydrogen. For instance, acetic acid yields free carbon, benzene, naphthalene, acetone and permanent gas, whilst CH_4O gives permanent gas, containing acetylene and other bodies.

The size of the retorts, likewise, has a slight effect on the yield of the products. This is more marked near the end of the distillation, when the heat of the retort is, as a rule, at its maximum; and the gas travels slowly, both through the heated mass and along the heated walls of the retort.

The aim of the wood distiller is to obtain the largest possible yield of acid; and he, therefore, regulates the heat of his retorts accordingly. Experience has proved that the most suitable temperature is 350° to 400° C.

As soon as the temperature exceeds 400° C., the per cent. of acetic acid decreases; and as acetic yields acetone on being passed through a red-hot tube, one would imagine that the per cent. of acetone would increase; but it is not the case. I made two trials, conducted under exactly the same conditions, with the exception that in one case the temperature was 300° to 350° C., and in the other low red-heat. The crude acid, from experiment at the low temperature, gave ·42 per cent. of acetone, and at the high temperature ·39 per cent. The yield of both acid and naphtha were, in these experiments, one-third less in the case of strong heat.

It is impossible to formulate the reactions which take place inside the retort, as you cannot establish a fixed set of conditions as regards temperature, etc. Certain compounds are no sooner formed, we will say in the centre of the retort, than they pass into a more strongly heated space, and are at once dissociated.

I have seen it stated that a sharp strong heat gives a better yield of naphtha, but my experience does not bear out this statement.

I now come, 3rdly, to speak of the preparation, properties and uses of wood naphtha.

We will assume that the wood has been distilled, and that we have the liquid distillate, containing acid, naphtha and tar, to work up for the naphtha. It is allowed to stand for some time, when the tar falls almost completely to the bottom of the vessel, and the crude acid forms a watery layer on the surface. This latter, amounting to 42 to 47 per cent. by weight of wood distilled, is drawn off and worked up for the acid and naphtha it contains, according as to what acid products are required. In this district these are, for the most part, acetate of lime, iron liquor, and brown sugar of lead.

If acetate of lime is wanted, the crude acid gets in most cases a plain distillation, to rid it of most of the tarry matters. The distillate is then neutralised with lime, allowed to settle, and the supernatant liquor distilled. The naphtha being the most volatile, comes over at the commencement, and should be collected until the runnings have a sp. gr. ·995 to 1·000. A very common test in use at many works is for the stillman to throw a small quantity of the runnings upon some hot cinders, and if the gases evolved do not take fire, he concludes it is time to put off the still. It is needless for me to dwell on the unreliability of such a test, and in these days, when we hear so much about the great strides which are being made in methods of testing in chemical works, it should be replaced by a better one.

If sugar of lead or iron liquor are wanted, the crude acid is distilled, and the naphtha collected, until the runnings show same strength as in the previous case. Care is required to keep the acid from coming over. I have often worked crude naphtha, which would have paid much better to work for the acid than for the naphtha it contained; but I find dull times are the best remedy for such carelessness.

The crude naphtha, by either process, has an average sp. gr. 0·980, and varies from 0·970 to 0·990. It has a dark-brown colour and strong smell, and must, from what has previously been said, be a complex liquid.

Besides water, crude wood naphtha contains the following compounds:—

	Formula.	Boiling. Point.	Sp. Gr.
Methyl Alcohol	CH_3OH	65·0	0·79589 at 15·56° C.
Acetone	$(CH_3)_2CO$	56·3	0·8144 at 0° C.
Methylic Acetate	$C_2H_3O_2CH_3$	56·3	0·90085 at 21° C.
Aldehyde	C_2H_4O	20·8	0·8009 at 0° C.
Allyl Alcohol	C_3H_5OH	103	0·8706 at 0° C.
Di-methyl Acetate	$C_2H_4\left\{\begin{matrix}OCH_3\\OCH_3\end{matrix}\right\}$	63-64	0·8757 at 6° C.

Also small quantities mono-, di-, tri-, methylamine, acetate of ammonia, oils and tarry matters.

The wood naphtha from manufacture of sugar of lead and iron liquor almost always contains free acetic acid.

There are several methods in use for working up the crude naphtha; but the one which finds most favour in this district, and which gives a very good finished product at a small cost for plant and material, is to distil with lime, which retains the water and acid, decomposes almost entirely the methylic acetate, simple and compound ammonias, acts upon the oils in such a manner as to convert the greater part of them into solid resinous bodies of high boiling points, and has also the effect of diminishing the percentage of acetone, which, on treatment with lime, gives condensation products of high boiling points. It is important to decompose the methylic acetate, as on account of its high sp. gr. it, if present in large quantities, makes it difficult to work the naphtha down to the usual strength, 61 o.p., sp. gr. ·827.

A source of considerable trouble to the rectifier of wood naphtha is the oils contained in the crude, and although they, for the most part, boil considerably over 100° C., on account of the large quantity of steam given off, are, in the distillation, easily carried over along with the naphtha. They are very soluble in strong naphtha, but can be separated again by merely diluting with water, when they usually rise as an oily layer to the surface of the liquid; in some cases they sink to the bottom of the vessel.

It is essential to remove these oils as completely as possible, as a small percentage is injurious, as I shall show farther on.

As to the composition of these oils very little is known. According to Cahours they contain, besides other bodies, toluene, xylene, and cymene; Kramer and Grodski found subsequently that they contain ketones, which, on treatment with $ZnCl_2$ and H_2So_4, give toluene, xylene and cymene.

I made a fractional distillation of a sample, and below you have the results:—

300cc. taken, sp.gr. ·876 at 15·5° C.
A: Thermometer in liquid.
B: ,, ,, vapour.
Began run: A, 80° C.
,, ,, B, 65° C.

	A.	B.
5%	90° C.	70° C.
10	110	84
20	133	120
30	140	130
40	146	136
50	152	142
60	160	151
70	168	158
80	178	168
90	193	183
95	203	191

Specific Gravity of the Distillate.

1st.—50cc.	0·840 at 15·5° C.
2nd.—50cc.	0·857 ,, ,,
3rd.—50cc.	0·872 ,, ,,
4th.—50cc.	0·881 ,, ,,
5th.—50cc.	0·887 ,, ,,
Last.—25cc.	0·901 ,, ,,

You will no doubt notice that in the case of B., a considerable proportion of the oil comes over 130-142. Within these temperatures the three isomeric xylenes boil. This fraction is almost completely absorbed when shaken with 1½ times its volume of strongest sulphuric acid, which would be the case with meta- and ortho-xylene, as they are converted into sulphonic acids, but not if it consisted of paraffins or para-xylene. I do not say that this property of the oil proves the presence of the above-mentioned hydrocarbons, but it tends in the right direction. The finished wood naphtha, as it comes into commerce, is an almost colourless liquid, having a sp. gr. of 0·827 at 15·5° C. This sp. gr. corresponds to 61 o.p. by Sykes.

One of the principal uses of wood naphtha is in the manufacture of methylated spirit, but there are certain conditions which must be fulfilled before the Government will allow of its being used for this purpose, one of which is that it must have a pungent smell, so that it can impart the same to the spirits of wine, and so destroy its "magic" influence—*i.e.*, render it unfit for drinking purposes.

The following is the clause in the Excise regulations which treats of the strength and proportion of wood naptha to be added to the spirits of wine for manufacture of methylated spirit:—

"The spirit is not to be less than 50 o.p., nor less in quantity than 450 gals. at a time, and the proportion of naphtha or methylic alcohol, not less than one-ninth of the spirit. Strength of naphtha to be at least 60 o.p."

The buyer always specifies that the naphtha shall be "miscible,"—*i.e.*, it shall not produce turbidity when diluted with water. This turbidity arises from the presence of the oils above referred to, and which, on addition of water, are thrown down in a finely divided state, and give the solution a milky appearance.

The method adopted in this district, and also by a large number of firms in England, for taking the strength of the finished naphtha, is simply determinative of its sp. gr. The instrument used is that of Sykes, the readings of which are tabulated with corresponding strengths. These tables were made up for spirits of wine, and are still used in all the

Government offices for that purpose, but it certainly seems a little strange that the strength of wood naphtha, consisting chiefly of methyl alcohol, should be fixed by them. It so happens that the sp. gr. of methyl and ethyl alcohols do not, for the same percentage, differ very much; but, as it is becoming more the custom to buy by sp. gr., corresponding to a certain percentage, it would be decidedly preferable to adopt a table made up for methyl alcohol. The Sykes table gives the highest strength at 60° F. to be 67 o.p., which corresponds to sp. gr. ·815, but this only corresponds to 93 per cent. of CH_4O, as determined by Prof. Dittmar and myself, and you can see that by still adhering to the old tables confusion may often occur. A good average sample of finished naphtha, sp. gr. ·829, at 15·5, contains—

CH_4O .... = 80%
Acetone .... = 6
H_2O .... = 12

Besides traces of methylic acetate, di-methyl acetol, methylamine and other impurities, should boil as under:—

100cc. taken.
A: Thermometer in liquid.
B: ,, ,, vapour.
Began to run: A, 67° C.
,, ,, B, 64° C.

	A.	B.
5%	67° C.	66° C.
10	67·5	67
20	67·5	67
30	68·0	67·5
40	68·5	68.0
50	69·0	68·5
60	70·0	69·0
70	70·5	69.5
80	72·0	71·0
90	76·0	75·0
95	84·0	79·0

Not very many years ago wood naphtha was scarcely a saleable article. Its principal uses were for varnish and finish manufacture, and as a substitute for methylated spirit; but with the discovery of the aniline colours, and the large increase in the consumption of methylated spirit, the demand for this article has increased, and to-day, instead of wood naphtha being cheaper than methylated spirit, it is the reverse, the former varying from 4s. to 5s. per gallon, and the latter from 2s. 6d. to 3s. per gallon.

The principal uses for finished wood naphtha are for methylated spirit, aniline colour and varnish manufacture, the portion rich in acetone being chosen for the latter purpose, as it is a capital solvent for gums.

The quantity used for aniline colour manufacture is very small in this country compared with Germany, but the quality required for that purpose is much higher than that employed in making methylated spirit. It is to be hoped that organic chemistry will be more taught and studied in England and Scotland, so that the manufacture of articles of commerce, such as aniline colours, will be thoroughly understood and appreciated both by young chemists and manufacturers.

DISCUSSION.

Mr. Beilby paid a tribute to the author for his valuable contribution. He felt that it was the duty of each particular Section to bring into discussion the industries of the locality; and testified to the great want hitherto felt of technical literature bearing upon these industries, a want which such papers were intended to supply. He would like to ask the author whether stove-drying of the wood had been found to have any beneficial effect upon the yield of products? He also wished to know what method was used to determine the percentage of methyl alcohol present in purified-wood spirit.

Mr. Fawsitt, in reply, said that stove-drying had not proved beneficial as regards increase in the yield of products. They always used the methyl-iodide test for methyl alcohol in purified-wood spirit.

In reply to questions asked by Mr. Pattison, he said that naphtha was made miscible in water by extracting the oily portions by distillation, and that its so-called solvent action upon gums and resins was due to the acetone present.

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Meeting held April 7.

MR. E. C. C. STANFORD IN THE CHAIR.

A NEW SYSTEM OF COOLING OILS FOR THE EXTRACTION OF PARAFFIN.

BY GEORGE BEILBY.

The separation of the solid from the liquid paraffins in their manufacture from crude shale oil or petroleum, is essentially a process of crystallisation and filtration. By fractional distillation of the partially purified oil a concentrated solution of solid paraffin in oil is obtained. In the manufacture as ordinarily practised in Scotland, this concentrated solution of paraffin, whose crystallising point is from 90° to 100° F., is exposed to the air in large trays or tanks, from one foot to two feet deep. In from two to four days, according to the season of the year, the oil is reduced to the temperature of the air or nearly so. The separation of paraffin by such slow crystallisation makes the solution into a perfectly solid mass, so thoroughly knit together by the interlacing of crystals, that the oil, which constitutes about 75 per cent. of the whole, cannot be separated by mere draining. In order that the oil and paraffin may be separated by filtration, it is necessary to crush the mass so as to destroy the general structure, leaving the individual crystals free. The crushed mass is pumped into filter presses, the oil passes through and the paraffin is retained in the cells. The paraffin obtained at this stage is known commercially as "hard scale," but a further crop of crystals of lower melting point is obtained from the first mother-liquor by concentrating it by distillation, and again cooling and filter-pressing. As this second crop of paraffin is of lower melting point, and is, therefore, more soluble in oil, it is the custom to cool the solution by means of artificial refrigerators, so as to crystallise and extract the paraffin at a temperature of 32° F., or as much below that point as possible. The apparatus most commonly used for this purpose consists of a drum or cylinder of cast-iron, kept at a low temperature by a current of cold brine led through it from a refrigerating machine. This drum revolves on a horizontal axis, its lower side dipping slightly in a tray containing the oil to be cooled. The cold surface of the drum, as it revolves, picks up a layer of oil from the tray, which layer, as it is carried round on the cold surface, is reduced to its temperature. After the layer has travelled round on the drum for about three-fourths of its revolution, it is scraped off by a steel knife which is pressed against the surface of the drum by springs and set screws. The cleaned surface in the course of its revolution again passes through the oil in the tray, taking up a fresh layer of solution to be cooled, solidified and scraped off. Thus the process is a rapid and continuous one, the warm oil is continually running into the tray, passing over the drum, and being scraped off and dropped into a suitable receptacle, from which it is drawn by pumps and forced into filter presses. Various modifications of this form of paraffin cooler have been designed by Mr. A. C. Kirk, Mr. Southby, Mr. N. M. Henderson, and Mr. William Baxter, in every case the object being the rapid cooling of a large quantity of oil. Owing to the very low conducting power of paraffin for heat, quick cooling can only be obtained by bringing the oil *in thin layers in contact with cold* metallic surfaces; this principle was recognised and acted on in all of the coolers enumerated. Looked at simply as appliances for cooling, the maximum quantity of oil through the maximum number of degrees in the shortest possible time, these coolers are all really efficient. It is only when the desired object is, not merely the cooling of a solution of paraffin to a given temperature, but the efficient separation of the paraffin by crystallisation, that the principle of rapid cooling in thin layers is seen to be unsound.

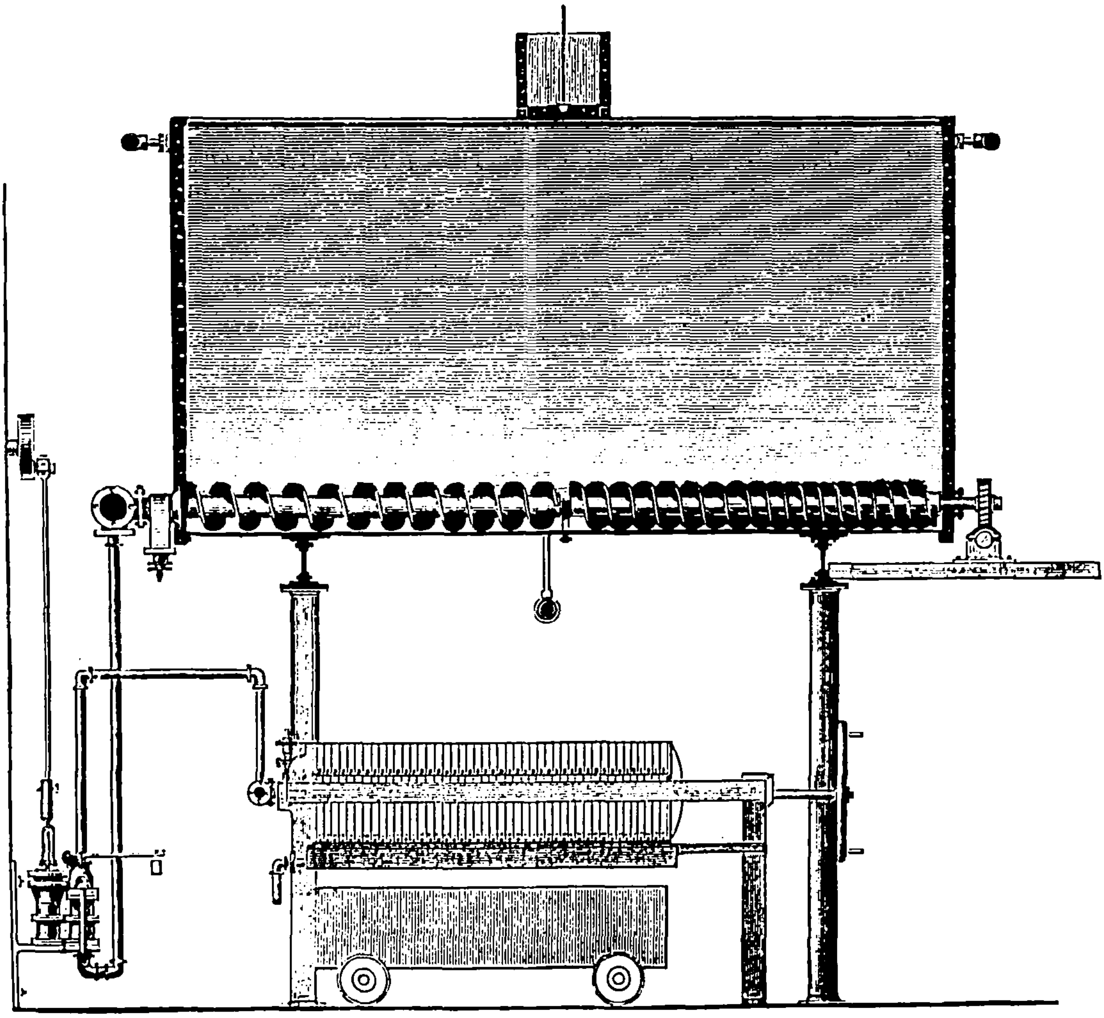

FIG. 1.

The crystallisation of paraffin from solution in oil is in many respects analogous to the crystallisation of salts from aqueous solution. The size of the resulting crystals in both cases is very largely determined by the rate of cooling and deposition. A hot saturated solution of sulphate of sodium suddenly cooled to the atmospheric temperature deposits the salt in a granular or powdery form, whereas slow regular cooling produces large well-defined crystals. In solutions of paraffin in oil these effects of cooling are particularly well marked. When a hot saturated solution is suddenly cooled, the paraffin is deposited in an amorphous or only faintly crystalline condition, and is consequently very difficult to separate from the oil. On the other hand, if the solution is kept in repose, and under such conditions that the cooling is slow and regular, the paraffin deposits in large well-defined crystals, which are easily separated from the oil. These effects are most apparent in oils of high viscosity, and for two reasons: (1.) The viscous oil on being cooled to a low temperature itself becomes gelatinous or semi-solid, and in this state tends to retain the paraffin in an imperfectly crystalline state. (2.) The methods of refining which are necessary for the production of the most viscous oils, also tend to produce viscous or amorphous paraffin. Vaseline when properly prepared shows no trace of crystalline structure, and is, therefore, an excellent example of an amorphous paraffin mixed with a viscous oil. As the whole tendency of paraffin and petroleum refiners is to produce lubricating oils of maximum viscosity, the difficulty of separating the paraffin tends also to increase. In certain cases the viscosity practically attainable is determined solely by the possibility of properly extracting the paraffin.

The value of slow regular cooling for the extraction of the first crop of paraffin or "hard scale" has for long been fully recognised, so far at least as natural or atmospheric cooling is concerned; but whenever refrigerator cooling has been used, the principle has been abandoned. Mr. A. C. Kirk tells me that at Mr. Young's Bathgate Works various schemes were proposed, and even partially worked out, for the cooling of paraffin oils in a state of repose, but these were put aside in favour of the more rapid system of cooling by drums.

At Oakbank, about two years ago, it became necessary to devise means for cooling and crystallising certain very viscous oils. An experience of many years with this class of oils made it evident that proper crystallisation could only be ensured by cooling the oil slowly, and in considerable mass, to the desired temperature. The success of Mr. Coleman and others in maintaining a winter temperature in the meat safes of ships in the Tropics by means of refrigerating machines, made it seem not unreasonable to consider the possibility of cooling paraffin oils in an artificial winter atmosphere. When ways and means came to be considered, it was evident that the employment of tray coolers, one to two feet deep, was quite out of the question, on account of the enormous ground area that would be occupied for a given quantity of oil, involving the erection and cooling of very large buildings. The quantity of oil to be cooled at Oakbank was 8000 gallons per day, and assuming that four days were necessary for cooling it, accommodation for four days' oil or 32,000 gallons would have to be provided. This quantity of oil spread out in tanks one foot deep would cover a ground area of 5120 square feet, equal to a rectangle of 160 feet by 32 feet.

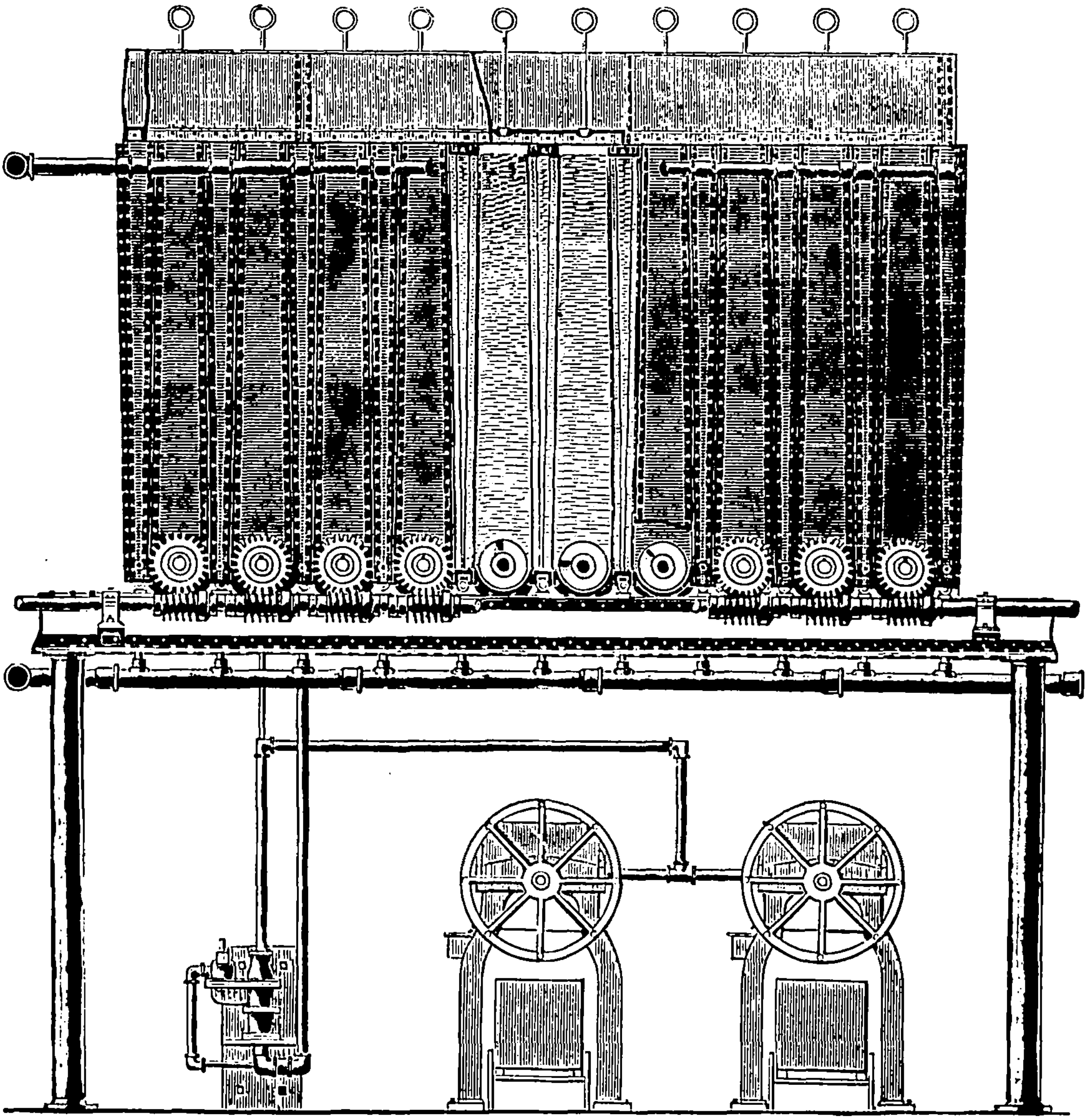

FIG. 2.

In the plans adopted and carried out at Oakbank, the coolers are turned edgewise, and are built in blocks with interspaces between the cells. It has been pointed out that the cooled and crystallised mass needs to be mashed or crushed before it can be filtered. In the flat tray coolers this has generally been done by manual labour, hand tools being forced through the mass. In the coolers of this new arrangement such treatment was out of the question; it was, therefore, necessary to provide mechanical appliances for crushing the mass, and delivering it ready for the filter presses. After experimenting with various forms of crusher, the arrangement finally adopted was a screw laid along the bottom of each cell. The shaft of this screw passes out through a stuffing gland on the end of the cooler, and is fitted with worm gearing, by which it can be rotated when desired. The thread of the screw is right-handed, so that by rotating it backwards, or from right to left, the block or slab of cooled paraffin is bored out, the "borings" or crushed paraffin being delivered through an opening at the opposite end from the driving shaft. As the screw occupies the whole length and breadth of the cell at the bottom, the whole base of the block of paraffin is sheared away by its rotation, and as the cooling cells are wedge-shaped—wider at the bottom than the top—the whole weight of the block rests on its base. As the base is sheared away the whole block settles down, and is gradually sheared and delivered by the screw.

The drawings (Figs. 1 and 2) show a block of cells in longitudinal and cross section. The interspaces between the oil cells are enclosed and provided with pipes for the circulation of cold brine.

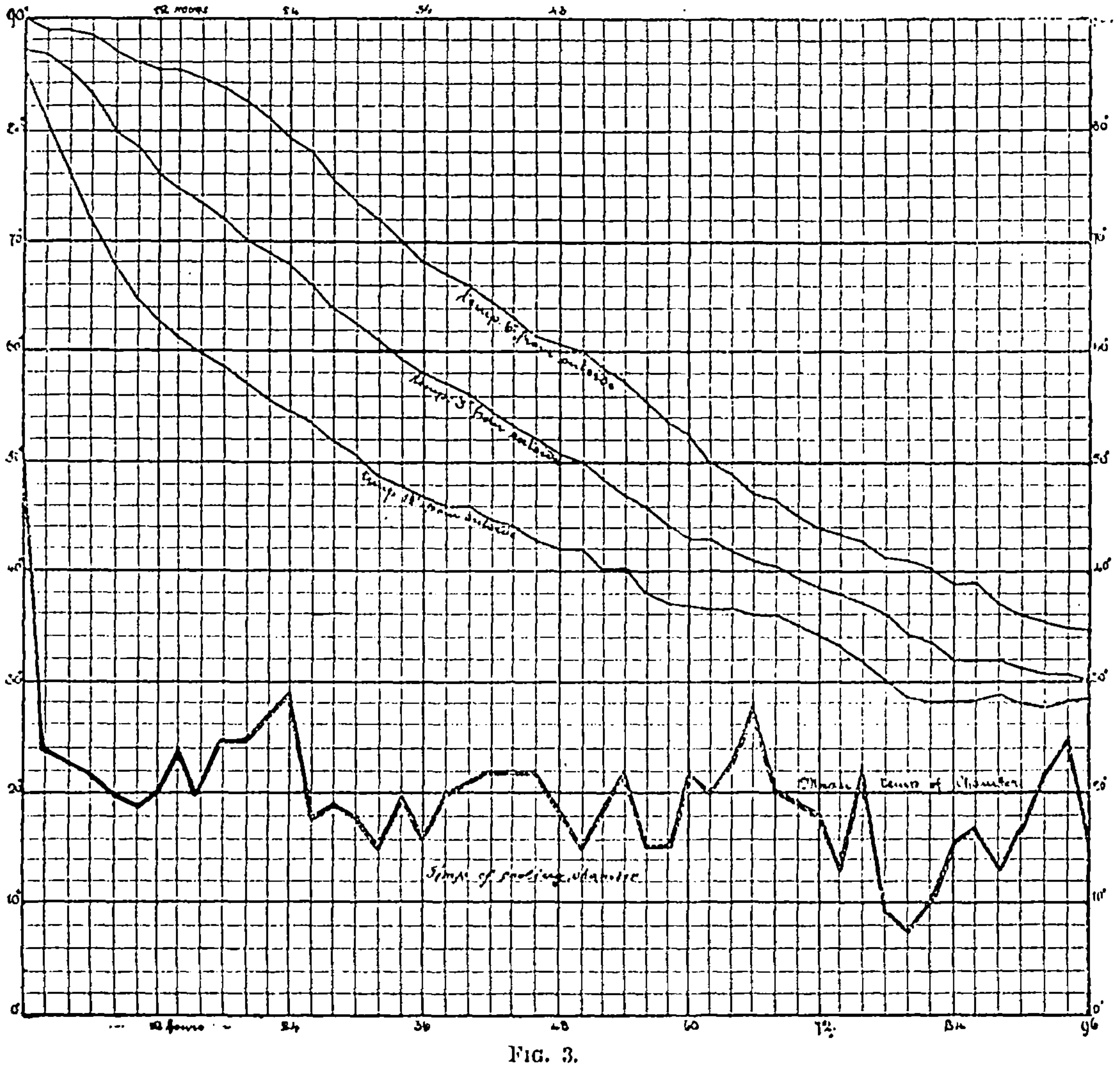

FIG. 3.

In designing this plant it was necessary to know exactly the time that must be allowed for the effective cooling and crystallisation in cell coolers of a given size and thickness. Experiments were made in a temporarily fitted-up cold air chamber. A cooling cell one foot thick was filled with paraffin solution, at 95° F., and was subjected in this cold air chamber to a mean temperature of 21° F. for 96 hours. In the cooling cell were placed thermometers 1½ inches, 3 inches, and 6 inches from the outside. The temperature indicated by these, and by another thermometer hanging in the chamber, were observed and recorded every two hours. The results of these observations were laid down on a diagram (Fig. 3). The great irregularity of the temperature of the chamber was due to the imperfect character of the cooling engine, and to the small size of the chamber, but fortunately the low conducting power of the oil and paraffin saves the mass from any great irregularity in cooling. The conclusions from these experiments were: That with a suitable cooling medium, oil and paraffin could be cooled sufficiently in 96 hours in cooling cells one foot in thickness; further, that with this rate of cooling the crystallisation of the paraffin was thoroughly satisfactory.

To aid in determining the needful dimensions and strength of the screw, gearing, etc., determinations were made of the crushing and shearing strength of the cooled block of paraffin at 32° F. These of course must vary with the percentage of paraffin and the viscosity of the solvent oil; it is, therefore, hardly worth while to reproduce them here. With the particular oil experimented with, it requires a pressure of three tons exerted on the pitch line of a 12-inch worm wheel to start the screw in the midst of the mass of cooled paraffin. The screw revolving at ·6 revolutions per minute throws 600 to 800 gallons of mashed paraffin per hour.

The cooling cells are arranged in four blocks of ten cells each. Each block contains one day's oil, or 8000 gallons. The blocks are carried on girders 10 feet above the floor level, the necessary filter presses being arranged on the floor below.

The house in which the cells are placed is built with double walls and a double roof, the interspaces being filled with non-conducting material.

Cold brine from an ammonia refrigerator is led through between the oil cells. A cold surface of about 1000 square feet is exposed to the air of the house, so that a considerable part of the effect of the refrigerator is devoted to keeping the air of the house cool. From experimental determinations of the conducting power of brick, it is estimated that the heat passing into the house through floor, walls, and roof, will amount during the heat of summer to the equivalent of six tons of ice melted per day.

DISCUSSION.

Mr. COLEMAN quite endorsed the author's views in respect of the advantages of slow cooling, compared with the old method of cooling rapidly by a revolving drum as detailed in the paper. He thought that the plan invented by Mr. Beilby of cooling in cells, and breaking up the mass by screw motion from below, was as ingenious as it seemed effective, and far less calculated to destroy the large crystals of paraffin. He stated that the reason why cold-air machines were used so extensively for meat preserving was that chemicals could not be employed for the purpose for hygienic and other reasons. But in the case of paraffin these objections were non-existent, and it then became a mere question of convenience and economy what method of refrigeration should be adopted. In conclusion, he considered that a debt of gratitude was due to the author for his very practical paper, and looked forward with interest to the results likely to accrue to the paraffin oil industry from the adoption of Mr. Beilby's process.

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ON DESTRUCTIVE DISTILLATION.

BY PROFESSOR MILLS, F.R.S.

IN the present paper I propose to deal with three topics—viz. (1) The definite character, (2) The limiting temperature, and (3) The course, of destructive distillation.

(1).—DEFINITE CHARACTER OF DESTRUCTIVE DISTILLATION.—It is universally recognised that when such bodies as calcic hydrate or carbonate, hydric salicylate or potassic acetate, are destructively distilled, the results are of a very definite nature, and it is the custom to express these by exact symbolic equalities. The destructive distillation, on the other hand, of coal, shale, bone, wool, cellulose and the like, has always been vaguely regarded; as if it were of an indefinite character, and open to a vast variety of modification at the hands of the chemical inventor. It appears to me that all kinds of destructive distillation are equally definite when the evidence with regard to them is equally complete; and that they cannot be susceptible of indefinite variation, because the law of multiple proportions must apply, without exception, to every one of them. Some illustrations of this principle may be of interest to the section.

(a) *Coal.*—Most authorities are agreed that coal has been derived from more or less impure woody fibre or cellulose, $nC_6H_{10}O_5$, under the influence of heat, pressure, and time. The effect of heat is at first to dehydrate cellulose. By interpolation among Violette's well-known results on the heating of wood (*Ann. Ch. Phys.* [3], xxxii. 304), I find that $nC_6H_8O_4$ corresponds to a temperature of about 185°, and $nC_6H_6O_3$ to about 220°, in the absence of pressure; in presence of pressure, the latter temperature corresponds to $nC_6H_4O_2$. At a point somewhat below 430°, and without pressure, the residue has the composition, nC_6H_2O. The final stage nC_6 is probably not attained under ordinary experimental conditions.

According to these results, the composition and reactions of coal should turn upon the value of n, the losses of H_2O, and the collateral kinetic changes which, occurring in the course of these definite transactions, lead to the formation of isomeric (or even of polymeric) coals. The organic matter in coal or shale, if we agree to represent its composition by a formula, should correspond to an initial symbol nC_6 or $2nC_3$.

In constructing equations to represent the transformations of coal and other complex bodies I shall, for the sake of convenience, frequently employ collocations of symbols to indicate mean composition; it will be understood that these collocations are not intended to suggest separate chemical compounds.

Prof. W. Foster (*Proc. Inst. C.E.*, April, 1884) has completely analysed, and distilled at a high temperature, two samples of Yorkshire and one of Durham coal His mean results, apart from sulphur, nitrogen, pit-water and ash, correspond to the following relations:—

$$2\,C_{24}H_{18}O = 33C + C_{15}H_{34}O + H_2O.$$

	Organic matter in coal.	Fixed carbon.	Gas and tar.	Organic water.
(Calc.)	100	61·5	35·7	2·8
(Found)	—	61·5	38·5	

Foster has also similarly examined a Scotch cannel. His data may in like manner be reduced as follows:—

$$2C_{12}H_{12}O = 12C + C_{12}H_{22}O + H_2O.$$

(Calc.)	—	41·9	52·9	5·2
(Found)	—	41·3	58·7	

In my work on Destructive Distillation (2nd Ed., p. 28) I have shown that the organic matter in a good average Scotch shale has almost exactly the composition $C_6H_{10}O$. According to additional data (*loc. cit.* p. 26), reduced as before, the changes which this undergoes at a low temperature may be represented by the equality,

$$7C_6H_{10}O = 18C + C_{24}H_{62}O_3 + 4H_2O$$

		Fixed carbon.	Gas and oil.	
(Calc.)	100	31·5	58·0	10·5
(Found)		31·2	58·3	10·5

At a high temperature, we have the relation

$$7C_6H_{10}O = 6C + C_{36}H_{62}O_3 + 4H_2O.$$

		Fixed carbon.	Gas and tar.	
(Calc.)	100	10·5	79·0	10·5
(Found)		12·8	76·0	11·2

The Heywood Cannel Gas Coal, which may be taken as representing an average Scotch cannel, has (*loc. cit.* and special communication to the author) been analysed and distilled. The reactions are—at a low temperature—

$$4C_9H_{12}O = 27C + C_9H_{46}O_3 + H_2O.$$

		Fixed carbon.	Gas and tar.	
(Calc.)	100	59·6	37·1	3·3
(Found)		58·1	38·3	3·6

and at a high temperature—

$$4C_9H_{12}O = 24C + C_{12}H_{46}O_3 + H_2O$$

		Fixed carbon.	Gas and tar.	
(Calc.)	100	52·9	43·8	3·3
(Found)		52·5	43·9	3·6

The results for Boghead coal are as follows :—At a low temperature—

$$3C_{12}H_{20}O = 15C + C_{21}H_{58}O_2 + H_2O$$

		Fixed carbon.	Gas and oil.	
(Calc.)	100	33·3	63·3	3·3
(Found)		33·3	64·1	2·6

At a high temperature—

$$3C_{12}H_{20}O = 6C + C_{30}H_{58}O_2 + H_2O$$

		Fixed carbon.	Gas and tar.	
(Calc.)	100	13·3	83·3	3·3
(Found)		12·8	84·6	2·6

There is thus considerable evidence in favour of the definite character of the destructive distillation of coal and shale. It is probable that the organic matter in these minerals can be always represented with an nC_3 formula; and that a very simple relation exists between the C of the fixed carbon on the one hand, and the C of the [gas and tar (or oil)] on the other. It is clear also that C_3 is the fundamental unit, or stable condition, in these effects; and that not less than this unit must be removed from, or left in, the "fixed carbon" during destructive distillation. Hence it is hopeless to expect that, for instance, more than fractional variations in the yield of oil from a shale can be effected by modifications in modern retorting.

It would have been a great gratification to me if I could have rendered this part of my subject more complete, by splitting up the symbolic collocations for "gas and tar (oil)" into two others, for gas and tar (oil) respectively. Unfortunately no data at present exist for this purpose.

The following reductions may, however, serve as a guide and assistance in future investigations.

P. Frankland (*Journal* iii. 273) has given the composition of fifteen samples of purified coal-gas (presumably bituminous). The mean percentage and variations are as follows :—

Ethylene and its equivalents.	Hydrogen.	Carbonic Oxide.	Marsh Gas.
6·69	47·39	4·05	38·83
1·45	3·74	0·69	1·57
Variation.	Variation.	Variation.	Variation.

These ratios correspond to

$$3C_2H_4 : 24H_2 : 2CO : 20CH_4 : 2CO_2$$

(=102vols.; the last being added to fulfil the condition of unpurified gas). Summing the above formulæ, we have the collocation $C_{30}H_{140}O_6$, which may otherwise be expressed as $30CH_4 : 6H_2O : 4H_2$. Having regard to variation, we shall be correct in regarding average unpurified coal-gas as consisting of redistributed marsh-gas and water. Here also the C_3 unit recurs.

The same author (*loc. cit.*) gives the composition of three samples of gas from Scotch cannels. His figures tend to show that this gas tends to consist of redistributed methyl and water; but the data are too few, and their variation too great, to allow of any very exact inference.

The following complete analyses of London and average cannel gas-tar (Scotch) have been made in my laboratory :—

	London.	Scotch Cannel.
* Carbon	77·53	85·33
* Hydrogen	6·33	7·33
* Nitrogen	1·03	·85
Sulphur	·61	·43
Oxygen	14·50	6·06
	100·00	100·00

If we deduct nitrogen and sulphur, we shall obtain the following results :—

	London.	$C_{21}H_{22}O_3$.	Scotch.	$C_{18}H_{20}O$.
Carbon	78·82	78·26	86·44	85·71
Hydrogen	6·44	6·83	7·42	7·94
Oxygen	14·71	14·91	6·14	6·35
	100·00	100·00	100·00	100·00

The ubiquitous C_3 unit again appears in the mean composition of the tars; it must therefore be common to both kinds of gas.

(β) *Cellulose.*—The material used for this experiment was well scoured handkerchief cloth. It contained 5·99 per cent. of water, and yielded ·65 per cent. of ash. The still consisted of a glass flask holding 1130cc., whose neck sloped slightly downwards. By means of a piece of combustion tubing about 1·3m. long, this was connected with a two-necked receiver, on the outside of which cold water was constantly playing. The top of the still was covered with sheet asbestos. Heat was applied by means of a Fletcher burner. The distillation lasted six hours, during which a red heat was gradually attained. The following are the particulars of an experiment (substance taken, 100grm.) :—

	Grammes.
Water measured after drawing from receiver	42·6
„ in substance	6·0
	36·5
Hydric acetate (sp. gr. 1·06) in water	2·4
	34·5
Acetate : water : : $C_2H_4O_2 : 2H_2O$	1·2
	35·7
Experimental drainage correction	1·1
Total water	36·8

=39·4 per cent. on dry organic cotton.

Fixed carbon in retort	26·5
Ash correction	·7
	25·8

=27·6 per cent. on dry organic cotton.
Tar (heavier than water), about 2·5cc.
Hydric acetate, 2·040grm.
=2·20 per cent. on dry organic cotton.

The equation is

$$3C_6H_{10}O_5 = 12C + C_6H_8O_4 + 11H_2O.$$

	Fixed carbon.	Gas and tar.	Organic water.
(Calc.) 100	29·6	29·6	40·8
(Found)	27·6	30·0	39·4

In this case the weight of (gas and tar) is about equal to that of the fixed carbon. The C_2 from the acetate, added to the tar, amounts to about 3·6 per cent. Hence the gas, saturated with moisture, must have amounted to about 26·0 per cent. [In this and similar experiments, all the acid in the distillate is reckoned acetic.]

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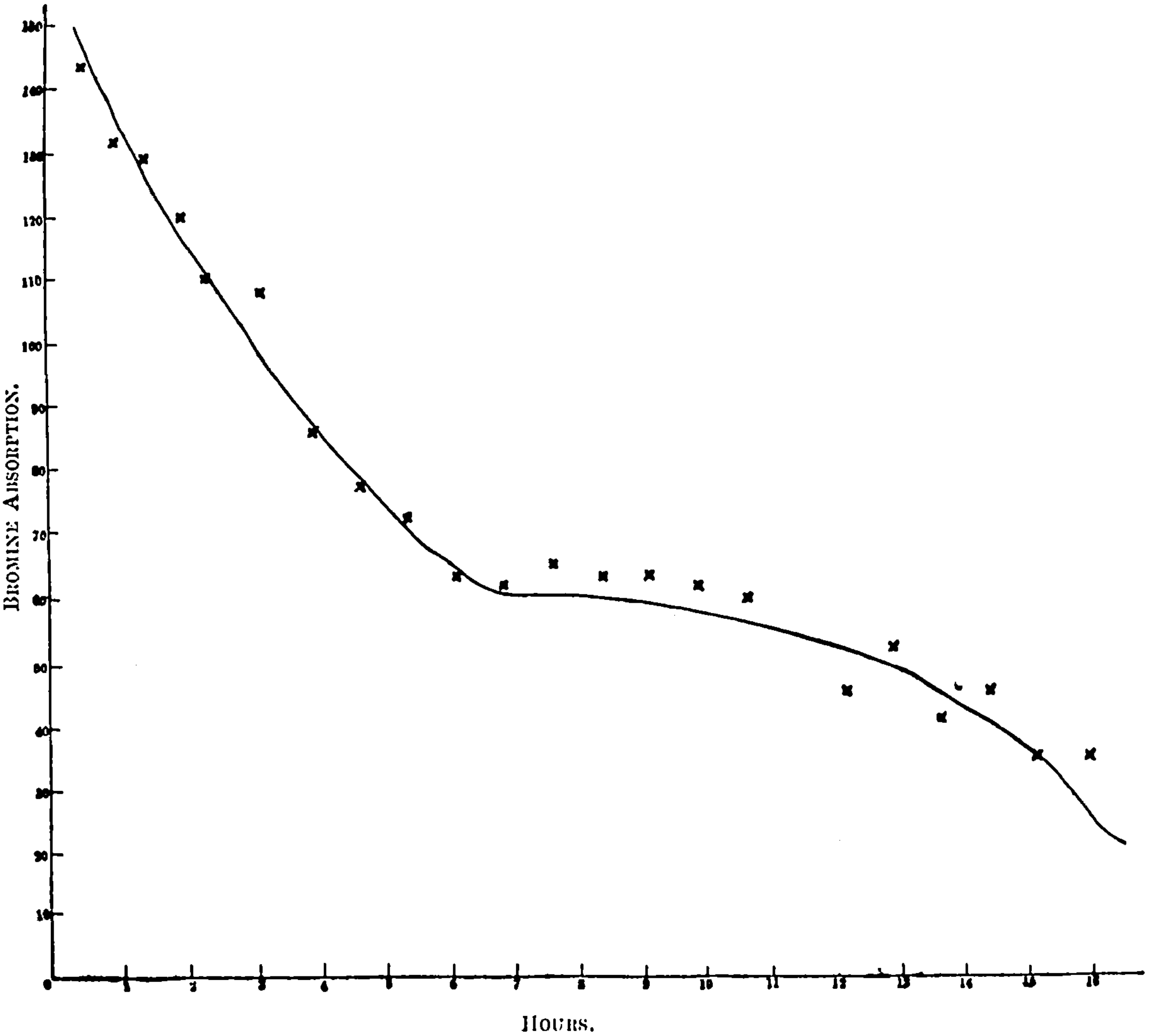
* Mean of two determinations.

(γ) *Jute.*—The sample contained 10·65 per cent. of water, and yielded 1·29 per cent. of ash. It was distilled in exactly the same way as the cellulose. The results, reduced as before, are in accordance with the relation :

$$C_{12}H_{18}O_9 = 7C + C_5H_8O_4 + 5H_2O.$$

	Fixed carbon.	Gas and tar.	Organic water.
(Calc.) 100...	27·5	43·1	29·4
(Found)......	27·5	41·3	31·2

The formula for jute is calculated from the analysis of Cross and Bevan (*Trans. Chem. Soc.* 1882, 100-101), who regard it as having the constitution of an aromatic cellulide. This may account for the unusual relations between the coefficients of C on the right-hand side of the equation. Jute furnishes 3·0 per cent. of acetate when distilled as above described. The amount of tar from 100 grammes exceeded 1cc. (a little having been lost). The gas, as before, may have been 38·8 per cent.



(δ) *Cannose.* — The sample contained ·15 per cent. of moisture, and yielded ·03 per cent. of ash. The same apparatus was used as before. The operation is extremely liable to fail, owing to intumescence. Accordingly, only 25 grammes were distilled, the time taken being eleven hours. The corrected results were as follows :—

$$C_{12}H_{22}O_{11} = 9C + C_3H_2O + 10H_2O.$$

	Fixed carbon.	Gas and tar.	Organic water.
(Calc.)	31·6	15·8	52·6
(Found)......	31·5	17·7	50·8

Sugar furnishes 2·42 per cent. of acetate when thus distilled, and very little tar. The gas probably amounted to about 17½ per cent. The C_3 unit now reappears.

(ε) *Wool.*—The sample, which consisted of well-scoured yarn, contained 14·93 per cent. of moisture, and yielded ·84 per cent. of ash. 50grm. of it were distilled during six hours in the same manner as before; excepting that a suitable tower, containing standard acid, was placed to intercept any ammonia that might pass off with the gas. The first products observed in the course of the distillation were hydric sulphide and water ; crystals of ammonic carbonate were observed next ; and these were succeeded by a pale yellow oil. The distillate smelled very strongly of members of the pyridine series; and a very pungent body, probably acridine, occurred in the latest stages of the operation.

Analyses of wool by Märcker and Schulze, and Scherer, are given in Watts's *Gmelin*, xvii. 351 [an unfortunate exchange of *H* for *N* in this analytical statement led me some time since (*Trans. Chem. Soc.* 1883, 142) to an erroneous formula for wool]. Their percentages agree fairly well with the expression adopted below.

	Märcker and Schulze. (Mean.)	Scherer.	$C_{39}H_{63}N_{11}SO_{13}$
Carbon	49·54	50·65	50·49
Hydrogen	7·29	7·03	7·01
Nitrogen	15·60	17·71	16·61
Sulphur	3·44		3·45
Oxygen	24·13		22·44
	100·00		100·00

The "fixed carbon" contained a considerable quantity of nitrogen. The following equation corresponds with the determinations so far as made:—

$$\underset{\text{"Fixed carbon"}}{C_{39}H_{63}N_{11}SO_{13}=C_{21}N_3}+\underset{\text{Gas and tar,}}{C_{10}H_{30}}$$

$$+5(NH_3CO_2)+3HCN+H_2S+3H_2O.$$

	Percentages found.	Calc.
Water	5·4	5·8
Residual carbon	24·9	26·1
Nitrogen therewith	4·3	4·5
Ammonia	8·6	9·2

It must, however, be added that the sulphur was not entirely evolved, as I found ·24 per cent. in the fixed nitrogenous carbon. Portions, also, of CO_2 and HCN ought doubtless to be credited to the gas and tar; but it would have been a matter of extreme difficulty to determine the free cyanide and carbonate. The actual tar amounted to about 2·5cc. per 50grm. of wool: it was lighter than water.

We have thus seen that as regards coal, cellulose, cannose and wool, the process of destructive distillation has certain stable stages; which, when expressed in the common chemical symbols, correspond to C_3 or a multiple of C_3. In the case of jute, also an apparently definite but partially aromatic compound, this relation either does not hold good or has become obscured. By adopting collocations of symbols to represent the average composition of the products of destructive distillation, we arrive at distinct and simple outlines of the complex results with which we had been previously familiar.

(2) LIMITING TEMPERATURES.—In a recent memoir [*Phil. Mag.* (5) xvii. 173] I have shown that the boiling points of all known normal paraffins having an even coefficient x of C are comprised in the equation

$$y=\frac{39\cdot315(x-3\cdot94)}{1+\cdot070753(x-3\cdot94)}$$

Similarly, when the coefficient of C is uneven, the equation is

$$y=\frac{38\cdot992(x-3\cdot92)}{1+\cdot070504(x-3\cdot92)}$$

When x is made exceedingly large in these equations, y (the boiling point) becomes 555°·67 and 552°·58 respectively. These values very nearly agree; and we may take their mean, 554°, as a working number. The normal paraffins have the highest boiling-points of any substances which it is the object of the shale-distilling industry to attain. Hence this number represents the highest temperatures required in the interior of a shale retort during the evolution of paraffins.

(3) THE COURSE OF DESTRUCTIVE DISTILLATION.—The course of destructive distillation admits of quantitative admeasurement in various ways. The usual method is to determine gravities; but so far as I am aware, no chemical method is ever systematically followed at works.

The destructive distillation of resin furnishes an excellent illustration of the ineffectiveness of the physical, as compared with the chemical, examination. While the extreme range of gravities in the distillate is only from ·90968 to 1·03038, the range of bromine absorptions is from 32·02 to 142·48. A distinguished firm of Glasgow distillers very kindly placed at my disposal a series of samples representing a complete distillation from one of their smaller stills. The samples were carefully sealed, and allowed to rest in a warm place for about eight months, at the end of which time the separation of the water was regarded as practically at an end. Two bromine absorptions (by the titration method) were then made for each sample, and the gravities determined at 9° C.

The resin used for distillation was American. It was blackish brown in colour. Specific gravity at 15° C. = 1·065; bromine absorption (determined colorimetrically) 101·66 per cent. [Pure resin absorbs nearly 112·96 per cent.]

The following table contains the whole of the results:—

No. of Sample.	Hours.	Sp. Gr.	Bromine Absorption.	Remarks.
			per cent.	
1	0·5	·90968	142·48	Spirit begins
2	1·0	·92308	131·56	
3	1·5	·92890	128·70	
4	2·0	·92342	119·24	
5	2·5	·93863	109·62	
6	3·25	·95100	107·18	Spirit ends. Oil begins
7	4·0	·98400	85·31	Not quite clear. More viscous
8	4·75	·98429	77·27	
9	5·50	·99601	71·20	
10	6·25	·99792	63·37	
11	7·00	·99820	60·74	
12	7·75	·99621	63·50	
13	8·50	·99621	61·28	
14	9·25	·99621	61·62	Darker coloured
15	10·00	·99332	59·61	Still darker
16	10·75	·99241	57·28	" "
17	11·50	·99181	52·60	Less dark
18	12·25	·99920	43·46	Dark layer at surface. Less viscous
19	13·00	·99880	50·76	Darkest of all
20	13·75	1·03038	38·83	Dark and turbid
21	14·50	1·01731	41·89	" "
22	15·25	·99122	32·30	Very dark
23	16·00	·96960	32·02	Nearly as dark as 19

From these experiments some interesting inferences may be drawn. In the first place it is evident that neither the specific gravity nor bromine absorption follows a perfectly regular course; this is very possibly due to unavoidable errors in firing, and to some superheating at the sides of the still, which did not contain more than about 1000 gallons. On the whole, however, the bromine absorption quite evidently decreases as the specific gravity increases, and from sp. gr. ·90968 to 1·03038 (the extreme range) 1 per cent. of bromine corresponds to about ·00058563 sp. gr. There is some indication of a break in the series, where spirit ends and oil begins. The extremely heavy bodies formed towards the close of the distillation split up at last into lighter ones; but as shown by the bromine absorption, these are probably of nearly the same chemical order as the heavy ones. As the course of the distillation proceeds from a great to a small bromine absorption, it involves the formation of more and more saturated bodies; in other words, an approximation is continually in progress towards the composition of the paraffin series.

In the accompanying drawing (page 327), I have plotted out the relation of bromine absorptions to time. Through the experimental points have been drawn two curves, whose equations are:

$$y = 158{\cdot}5({\cdot}86406)^t \text{ and}$$
$$y = 62{\cdot}4 - 2{\cdot}2657(1{\cdot}3689)^{t-7}$$

t being the number of hours, and y the bromine absorption. It will be observed that the first curve is in fair agreement with the actual work, and indicates that the bromine absoption could not exceed 158·5 per cent. This curve terminates at about the seventh hour, after which the absorption alters very little until the tenth hour, when it decidedly begins to fall. The exact position of the second curve is much more difficult to find, and lies less close to the experimental points. This indeed might have been reasonably expected from the diminished content of the still, the increasing effect of the heat, and the consequent magnifying of every irregularity that occurred. Better results could doubtless be attained with a still of greater capacity.

I wish to express my great indebtedness to Mr. Muter, and in particular to Mr. C. Ellis, F.C.S., for their practical assistance in connection with this paper.

DISCUSSION.

Mr. BEILBY, in proposing a vote of thanks to Professor Mills, said: It is unfortunate that Dr. Mills is not present to give us some further information as to the principles which have guided him to some of the deductions made in his paper. I am satisfied that the chemistry of destructive distillation can most profitably be studied if pure substances of known composition and constitution are operated on. Coal and shale are such complex substances, that even if their behaviour on distillation is carefully studied, the knowledge so gained can only contribute detached or partially connected facts, from which general laws cannot be deduced. I quite agree with Dr. Mills that the possible yield of paraffins from a given shale is quite definitely limited by the nature of the material, but are we further to understand him to say that these limits can be defined by a mathematical formula when nothing but the *ultimate composition* of the shale is known!

◆◆◆◆◆◆◆◆◆◆◆

A NEW PROCESS FOR THE ESTIMATION OF SULPHUR IN PYRITES.

BY JOHN CLARK, PH.D., ETC.

As pyrites in this country is usually sold on analysis, the correct estimation of the sulphur which it contains is a matter of very great importance, both to buyer and seller. Chemists are practically unanimous in their opinion, that the best way of estimating the sulphur consists in oxidising it to sulphuric acid, and afterwards precipitating it as sulphate of barium. The oxidation of the sulphur can be effected either by a wet or dry process. The wet methods which consist in oxidising the sulphur with nitric acid, nitro-hydrochloric acid, or chlorate of potash and hydrochloric acid, require great attention to details to obtain even approximate results; but even when the greatest care is taken there are always two sources of error due, first, to the iron which is carried down by the sulphate of barium, and, secondly, to the solubility of the sulphate of barium in the acid perchloride of iron solution.

These errors, it is true, operate in different directions, but they do not counterbalance one another, the increase of weight due to iron being as a rule small, compared to the loss arising from the solubility of the sulphate of barium. The existence of these errors has been clearly proved by the experiments of Fresenius (*Zeitschrift*, xix. 53), and is admitted by Lunge (*Zeitschrift*, xix. 419); and it is to them that the difficulty of obtaining concordant results is frequently due. That considerable differences may be expected, is very well illustrated in the valuable paper of Fresenius to which I have just referred, and in which he points out that four assistants operating on the same sample, and strictly in accordance with the details laid down by Lunge in his work on sulphuric acid and alkali, found respectively:

$$\left.\begin{array}{r}42{\cdot}14\\42{\cdot}46\\42{\cdot}76\\43{\cdot}10\end{array}\right\}\text{Per cent. of sulphur.}\left\{\begin{array}{l}\text{Mean}\\42{\cdot}61\end{array}\right.$$

or a difference of ·96 per cent. of sulphur between the highest and the lowest estimation. I do not think however that these results represent the capabilities of the wet method, in the hands of experienced analysts, as by a slight modification of Lunge's method very concordant results can be obtained, provided the operator uses always the same quantity of acid and works under the same conditions. By this means the error becomes less apparent, but it does not cease to exist. To avoid the sources of error to which I have referred, Lunge proposes to separate the iron by means of ammonia, before precipitating the sulphuric acid with chloride of barium; but I think it will be generally admitted that the elimination of the iron can be effected more conveniently by a dry fusion process.

For this purpose Fresenius recommends the fusion of the pyrites with ten parts of a mixture of carbonate of soda and nitrate of potash, extraction with water, precipitation of any lead with carbonic acid, evaporation with HCl to expel nitric acid, and precipitation with chloride of barium in presence of a small quantity of free acid. By this means the whole of the sulphur of the pyrites is obtained. The same sample of pyrites to which I have referred, when treated in this way gave in the hands of Fresenius' assistants:—

$$\left.\begin{array}{r}43{\cdot}82\\44{\cdot}04\\43{\cdot}74\end{array}\right\}\text{Per cent. of sulphur}\left\{\begin{array}{l}\text{Mean}\\43{\cdot}87\end{array}\right.$$

or a difference of 1·26 per cent. between the means of the wet and the dry method.

The objections to this process are (1) The liability to loss by spurting from the violence of the action which takes place when the pyrites is fused with a mixture of carbonate of soda and nitrate of potash.

(2) The difficulty of operating upon more than half a gramme of the substance.

(3) The time occupied in evaporating to get rid of the nitric acid.

The new method of estimating the sulphur in pyrites, which I have to lay before you, avoids these objections, and at the same time secures the elimination of the iron.

C. Fahlberg and M. W. Iles discovered (*Ber. der Deut. Chem. Ges.* xi. 1187) that when sulphides are fused with caustic potash or caustic soda, the sulphur is completely converted into alkaline sulphite or a mixture of sulphite and sulphate, provided at least twenty-five parts of alkali are used for each part of sulphur, and they pointed out that under these conditions no sulphide or hyposulphite was produced.

In the course of my experiments, I found that when the alkali is intimately mixed with calcined magnesia, the sulphur under favourable conditions can be completely converted into sulphuric acid or alkaline sulphate with little more than the theoretical quantity of alkali, but it is always advisable to use a moderate excess. It is upon this action that the process for the estimation of sulphur in pyrites which I am about to describe is based.

Process.—The magnesia mixture which I employ is made by grinding together, in a porcelain mortar, pure sodium hydrate made direct from the metal, and pure freshly-calcined magnesia. The product is a light powder, which can be kept for any length of time in a properly stoppered bottle, without undergoing any appreciable change. A blank experiment must always be made with each lot of the mixture to test the purity of the materials, and any sulphur found is deducted.

In carrying out the process, from 1 to 1½grm. of the dry pyrites, in a fine state of division, is introduced into a platinum crucible with four times its weight of the pulverised magnesia and sodium hydrate, and the whole is intimately mixed in the crucible itself, by stirring with a glass rod. The crucible is then placed in the mouth of a muffle furnace, and exposed to a low red heat for about three-quarters of an hour. Oxidation begins at once, and is practically complete in less than half-an-hour. The contents of the crucible do not fuse but simply contract in volume, and leave a space between the sides of the crucible and the contents, and there is practically no action on the platinum. After heating for three-quarters of an hour, the crucible is allowed to cool, then placed in a vessel containing water. In a few minutes the contents fall to powder, and the crucible is washed out. A stream of carbonic acid gas is then passed into the solution containing the insoluble, for about a minute, to precipitate any lead which may be present and carbonate the remaining alkali. The contents of the vessel are then boiled for a short time, and the solution passed through a filter. The insoluble is boiled three successive times with water, and about half a gramme of pure carbonate of soda, then thrown upon the filter and well washed with boiling water. The filtrate, which will now contain the whole of the sulphur as sulphate of sodium, and be perfectly free from iron and magnesia, is first neutralised with HCl, then acidified with about 3cc. of HCl, heated to the boiling point, and the sulphuric acid precipitated with a slight excess of chloride of barium. The precipitate is allowed to stand for several hours, then filtered, washed, dried, ignited, and weighed. The sulphate of barium thus obtained is perfectly pure. It is always advisable to dissolve the residue on the filter in HCl, and test for sulphuric acid. If any precipitate makes its appearance after standing for several hours, it must be filtered off and added to the result; but with proper care, this will be found to be merely a precautionary operation. I have made a great many estimations of sulphur in different samples of pyrites by this process, and have invariably got results which agreed very closely with one another. I have found, however, that to get complete oxidation with the above proportions of soda and magnesia, it is necessary to use the muffle, or at least to heat the crucible in such a way that the products of combustion will not come in contact with the contents of the crucible. Apart from the danger of the result being vitiated by the sulphur in the gas, the process of oxidation is affected by the products of combustion, so that it requires a much larger proportion of the magnesia mixture to effect complete oxidation.

For the purpose of testing this process, and comparing the results with those obtained by experienced analysts by the ordinary wet method, Mr. Inglis, who is the head of the laboratory of the Tharsis Sulphur and Copper Company, very kindly furnished me with samples of pyrites, which had been carefully analysed under his supervision, and I may add that I was not aware of the results, by the wet method, till my tests were completed and in Mr. Inglis's hands.

The samples were marked A and B, and the following are the percentages of sulphur which were obtained by both processes :—

	Clark's New Method. Sulphur per cent.			Tharsis's Wet Method. Sulphur per cent.	
	A.	B.		A.	B.
1 Exp.	50·18 ...	46·13	1 Exp.	49·46 ...	45·68
2 ,,	50·00 ...	46·17	2 ,,	49·58 ...	45·50
3 ,,	50·01 ...	46·16	3 ,,	49·58 ...	45·41
4 ,,	50·08 ...	46·18	4 ,,	49·62 ...	45·48
5 ,,	50·07 ...	46·16			
Mean	50·06	46·16	Mean	49·56	45·51

Another sample of pyrites which, in the hands of Mr. Tatlock, gave by the ordinary wet method 46·64 and 46·61 per cent. of sulphur, when analysed by my method yielded

47·24 per cent. of sulphur.
47·41 ,, ,,
47·20 ,, ,,
47·22 ,, ,,

or a mean of 47·26 per cent. of sulphur.

From these analyses, and a number of others which I could enumerate, if necessary, it would appear that the results obtained by this method are somewhat higher (about ½ per cent.) than by the wet method. This is what was to be expected when the sources of error to which I have referred are eliminated, and it corroborates the statements of Fresenius, although the difference is not so great as his experiments led me to expect.

The process which I have just described does not occupy more time than the ordinary wet method, and it avoids the sources of error—viz., nitrates, iron and excess of free acid—which so often lead to discrepancies.

DISCUSSION.

Mr. Tatlock testified to the importance of the subject brought before them by Dr. Clark. He felt that had the reading of the paper been the first intimation to him of the new process referred to, he would have felt sceptical of any advantages to be derived from it, especially in view of the concordant if indeed not absolutely correct, results of the wet process as shown by the table exhibited by the Tharsis Company. But having had an opportunity of examining the new process in detail, he felt convinced that it was destined to revolutionise the analysis of pyrites. At the same time he admitted the adoption of that process would inflict a certain hardship upon consumers, who, by a more complete estimation of sulphur in pyrites would have to pay for more sulphur without obtaining any additional amount. At the same time he hardly thought that it would be sufficient ground for repudiation of contracts. Chemists, however, had nothing to do with these matters; their sole concern was to render methods of analysis as perfect as possible.

The Chairman, in concluding the discussion, thought that Dr. Clark had proved his case. There could be no doubt that the old process was open to objection on the grounds stated, slight though these grounds might be. He sympathised with Mr. Tatlock's early scepticism as to the process, but thought that it was practicable if only due care were taken to employ absolutely pure reagents.

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The first meeting of the Section will take place on June 5th, when a Conversazione will be held at University College, Nottingham, on which occasion the Chairman will deliver his inaugural address.

Notices of papers and communications for the meetings to be sent to the Local Secretary.

Journal and Patent Literature.

I.—GENERAL PLANT, APPARATUS, AND MACHINERY.

Lixiviation Apparatus. H. Parenty. Revue Industrielle, 1884, 353.

This apparatus, illustrated by Figs. 1 and 2, consists of four cylindrical vats, each capable of holding about 60 litres of water, and 60kilos. of the material to be operated upon. The vats rest upon an oaken stand, and each is connected with the next one by means of a metal tube *a* leading into an upright tube *b*, open above and below. Each upright tube is provided with a valve below the metal connecting tube, in order to permit the connection between two adjacent vats being cut. A tube *d* at the bottom of each vat, and just beneath the upright tube, serves to empty the vat; a perforated plate at *c* acts as a strainer. A cistern A for water stands upon the four circular vats. It is fitted with a gauge and tube, also a U-tube, with graduations, to measure the duration of the flow of water. In the bottom are four openings corresponding in position with the vats. The apparatus is mainly an adaptation of the Buff-Dunlop black-ash vat system to smaller and more exact operations, or even for laboratory use.—W. D. B.

Fig. 1.

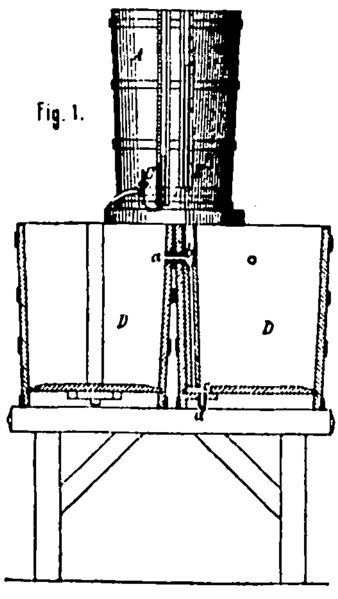
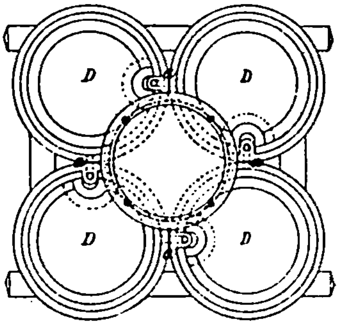


Fig. 2.



Improvements in Filter-presses. J. H. Porter and J. Porter, London. Eng. Pat. 6808, April 25, 1884.

The object of the patentees is to dispense with the filter cloths or strainers used in ordinary filter-presses. The plates are formed with a series of ribs upon their surfaces, which may be rectangular or angular. The projecting portions in one plate are opposite the spaces in the next plate, so that the passage of any liquid through the machine is diverted from one side to the other in a zig-zag fashion. The flow of liquid into the filter is continuous throughout its length, so that it passes over the whole surfaces of the plates in succession, inlets being formed alternately at the top and bottom of the chambers. The patentees state that the continuous friction of the surfaces of the plates as formed, causes the solid matter to be retained on such surfaces, the fluid being clarified thereby. Sand or charcoal may further be placed between the plates, so as to obtain further clarification. The claims are (1) the construction of filter-presses, in which the use of cloths is substituted by the friction of the surfaces formed as described; (2) filter-presses as described and shown.—C. C. H.

An Improved Filtering Apparatus. A. Domeier and B. Nickels, London. Eng. Pat. 7572, May 10, 1884.

The improved apparatus is intended for use with liquids which contain a very small quantity of solid matter. It consists of a cylindrical tank, fitted with a cover and manhole at the upper part; the lower end is provided with a perforated bottom above a receiving outlet chamber, and a second manhole on a level therewith for the purpose of cleansing. The usual pressure gauge, inlet cocks, etc., are provided. The perforated plate is covered with cloth, over which is placed a second perforated plate; on the top of this is placed the filtering bed, consisting of any suitable substance, such as charcoal, sand, sawdust, lime, etc. The liquid is pumped into the cylinder, and then forced through by compressed air, the operation being repeated until the filtering bed is stopped, when it is removed through the lower manhole, a fresh bed formed, and the operation of filtering again started. The patentees claim under two heads the apparatus as described.—C. C. H.

Improvements in Filter-presses. H. H. Lake, London. From R. H. Smith, Carbondale, Pennsylvania, U.S.A. Eng. Pat. 13,580, October 14, 1884.

THIS patent is for an improved construction of plate used in filter-presses. A circular plate is built up of wrought-iron plates, a solid centre disc and two perforated discs, one on either side. The rim of the plate is formed of canvas or duck, several thicknesses being stitched together to form the recess for the deposition of the solid matter; this is secured to the plate by rivets. To strengthen the plate, pads or bosses are rivetted on its surface, of the same material and thickness as the rim; three or more of such pads are used, and bearing against each other when the plates are in the machine, they form a series of supports, thus preventing the plates from collapsing. The several details of construction are covered by seven distinct claims.—C. C. H.

II.—FUEL, GAS, AND LIGHT.

Calorimeter for Fuel. Fr. Schwackhöfer. Zeits. Anal. Chem. **23**, 453.

THE author has constructed a calorimeter for determining the calorific power of coal or other fuel. The calorimeters usually employed are only adapted for small quantities of substance (a fraction of a grm.), and produce smoke, indicating thereby incomplete combustion. The author claims that in his apparatus these disadvantages are entirely avoided. The construction is given in the accompanying diagram.[*] A and A_1 are platinum combustion vessels, B a copper jacket, C the inner water vessel (the actual calorimeter) of nickled copper, D the inner non-conducting layer of eider-down, E a double-walled water vessel, F the exterior non-conducting layer of fine down, G a cylindrical wooden case, H the upper water vessel, J an annular cork board, K the lid, L the inner agitator, S and S_1 mirrors for observing the process of combustion, T_1 to T_{10} thermometers. The most important part of the apparatus is the combustion vessel. It is of platinum, and divided into two parts; the lower A is for the sample of coal, the upper A_1 for sugar charcoal. The chambers have openings at *a* and *b*, for introducing the fuel, and also for removing the sieves *c* and *d* after each experiment. The sieves are of platinum foil, perforated specially for the apparatus. The perforations of the lower sieve *c* are so small that they are only visible by transmitted light. In the upper sieve the holes are somewhat larger. Each is provided with an upright rim, which fits tightly to the side, and keeps it in its place. Three tubes are connected with the chamber A. *e* conducts oxygen from beneath the coal. The tube *f* has a double use: (1) to bring oxygen above the coal in order to ensure complete combustion of the evolved gases; (2) to observe the process of combustion by reflection from the mirror S_1. This is necessary, as the stream of oxygen requires to be carefully regulated, and the end of the combustion ascertained. The side tube *g* terminates under the sieve *d*. It is provided with a cross piece, and perforated on the upper side. It conducts oxygen to the upper chamber. All these tubes (*e*, *f*, *g*) are connected with well-fitting tubes, let into the lid of the case. After determining the actual thermal value of given volumes of water in the different vessels composing the calorimeter, it is next necessary to prepare and analyse quantitatively and calorimetrically the sugar charcoal to be used in the experiments. The calorimeter is then manipulated as follows: The vessel C is filled with 5200cc. of water at the temperature of the air, and 5·6grm. of the finely powdered sample of coal, and 2·4grm. of the charcoal weighed out. The quantities should be so chosen that the rise of temperature in C may be about 10° C. The combustion lasts about an hour. The coal is introduced into A, the charcoal into A_1, the two pieces then put together, and the jacket B screwed down, the lid is lowered, the thermometers inserted, and the mirrors mounted. When the apparatus indicates a constant temperature the combustion is commenced. Oxygen is led through *f*, the cap S raised, a small glowing chip thrown in, and the cap refitted. The charcoal in A_1 is kindled, and the aspirator O is put in action. The bottle N is then closed, and after some minutes the smaller aspirator is set going. The flow of water from O and P is regulated so that about five-sixths of the gases collect in O, and one-sixth in P. When the charcoal is almost burnt, sparks fall through the sieve and kindle the coal beneath. A quicker stream of oxygen is then passed through *f*, and introduced through *g*; towards the end of the experiment the oxygen through *g* is stopped, and *e* is opened. The combustion proceeds regularly, and not a trace of smoke appears in N. The combustion of both charcoal and coal should be complete. When the combustion is finished the aspirators are disconnected, the water is agitated by the mixer until the thermometers T_1 and T_2 stand at the same temperature, and the other thermometers are then read off. The volume of gas collected in the aspirators is noted, and the gas in P analysed. From these data the author has made a number of calorimetric determinations, which agree well together, and are somewhat higher than the value calculated by Dulong's formula from the percentage composition.—J. B. C.

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[*] For full details the original paper should be consulted.

An Improved Method of and Apparatus for Extracting Ammonia from Furnace Gases. W. R. Lake, London. From the Société Anonyme Lorraine Industrielle de Hussigny. Eng. Pat. 5898, April 3, 1884.

THE inventor recommends the absorption of ammonia derived from furnace gases by calcium chloride. The mixture of calcium carbonate and ammonium chloride formed is either to be used as an ammoniacal manure, or by heating sesquicarbonate of ammonia can be obtained, which is to be employed in the ammonia soda process. —S. H.

Improvements in Gas Producers and in Fuel-supplying Devices used in connection therewith. J. G. Wilson, Manchester. From J. E. Bott, U.S.A. Eng. Pat. 6764, April 24, 1884.

THE novelty of this invention consists mainly in the manner of admitting the air necessary for ensuring complete combustion of the fuel undergoing destructive distillation in gas producers, continuous corrugated tuyeres injecting air, or air and steam, being employed, which supply the fuel with a continuous blast of steam. This by means of the variable vertical section of the waved tuyeres causes the blast to permeate a larger vertical, as well as horizontal mass of incandescent fuel, thus ensuring a uniform combustion taking place, and the distillation of the fuel into gas proceeding regularly, whilst the residue is left in a more friable form than is possible when blast is admitted in the form of concentrated jets through isolated tuyeres, for in the latter case combustion proceeds irregularly, and the tendency is to form clinkers in the immediate vicinity of the tuyeres whilst the gas is produced in flushes.—D. B.

Improvements in the Purification of Coal Gas or of other Gases Containing Ammonia or Ammoniacal Compounds. A. P. Price. Eng. Pat. 6983, April 29, 1884.

THE purification of the gas is effected by means of the hydrogen sulphates, sulphites or phosphates of potassium, sodium, or ammonium, either in a divided condition or in solution, whereby the ammonia existing in the ammoniacal products contained in the gas is absorbed, and may be subsequently recovered from the resulting compound.—D. B.

Improvements in the Production of Combustible Gases from Finely-pulverised Coal, Peat, Wood-sawdust, or other Material. F. J. Jones, Worcester. Eng. Pat. 915, January 22, 1885.

This invention relates to the production of heating or illuminating gas, and consists essentially in passing a regular supply of finely-pulverised coal, peat, etc., through an hermetically closed chamber of suitable construction provided with the necessary inlets and outlets, and heated to a temperature sufficient for evolving combustible gases from the materials introduced.—D. B.

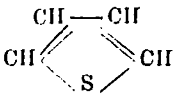
III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

Presence of High-boiling Phenols in Coal-tar. K. E. Schulze. Ber. **18**, 150.

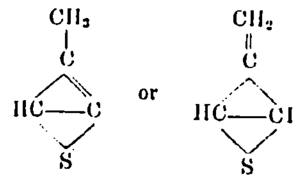
The author has isolated α- and β-naphthol from "green oil"—anthracene oil from which the crude anthracene has been extracted by filtration—and gives a detailed account of the process employed (*Ann.* 227, 143).—D. B.

Constitution of Thiophen Compounds. V. Meyer. Ber. **18**, 526-529.

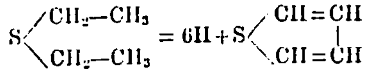
The essential points to be considered in proving the correctness of the formula



assigned to thiophen are (1) the number of isomeric substitution products of thiophen; (2) its property to form addition products; (3) the position of the sulphur atom; and (4) the behaviour of thiophen when subjected to oxidation. Referring to the position of the sulphur, which can be conceived to be combined in no other form than that of C–S or C–S–C or C–S–H, the author is of opinion that, taking into account the fact that thiophen is neither a thioketone, thioaldehyde, or mercaptan, there can no longer be any doubt as to the meaning expressed by its formula. The assumption that thiophen occupies the same position among its homologues as that held by benzene among the aromatic hydrocarbons requires, however, additional proof, which can be adduced only by taking into consideration the property which thiophen exhibits when subjected to oxidation. Although not yet finally decided, it is very probable that thiophen represents the first member of the homologous series, and little plausible as formulæ, such as—

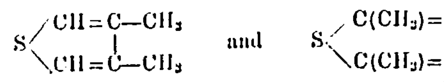


may appear at first sight, they have nevertheless not been hitherto confuted. As the homologues of thiophen are converted into thiophen carboxylic acid, when oxidised with potassium permanganate, it remained to be shown that when thiophen is similarly treated no acid is obtained which contains the same amount of carbon, or has the properties characterising a thiophen derivative. On oxidising pure thiophen with potassium permanganate, fatty acids and sulphuric acid were obtained. The substituting action of bromine and chlorine on thiophen is so energetic that the isolation of the resulting addition products is attended with great difficulties, and should the same be found to apply to the iodine addition products, the best mode of procedure will probably be to prepare the mellitic acid of thiophen—tetra-carboxylic acid—and reduce it with sodium amalgam. Should the resulting acid take up four atoms of hydrogen, this circumstance would confirm the assumption of two double unions, seeing that mellitic acid takes up double the number of hydrogen atoms compared with the number of double unions it contains. It is therefore intended to prepare a tetralkylthiophen and convert it into the acid in question by treatment with potassium permanganate. Referring to the number of isomeric substitution products, the author is of opinion that should it be shown that the mono-derivatives are obtainable in two modifications only, this again would prove the correctness of the formula for thiophen. In the preparation of the sulphonic and carboxylic acids of thiophen and of methylthiophen, only two modifications have as yet been observed, a circumstance which should however be verified by further research. It is of especial importance to obtain means for ascertaining the position of the substituted group in the thiophen molecule. In a previous communication, the formation of the anhydride of a disulphonic acid, and the assumption that dibromothiophen is analogous in constitution to para-dibromobenzene, formed the basis of the author's speculations. He is continuing his researches in this direction. The three dicarboxylic acids of benzene being distinguished by characteristic properties, it was thought that by studying the behaviour of thiophen dicarboxylic acids it might be possible to observe an analogy between them and the three phthalic acids, and thus determine the position of the carboxyl groups. At the author's instigation, Jackel prepared a dicarboxylic acid from the disulphonic acid according to Merz's reaction. An acid was obtained which, although not yet fully examined, appeared to be the terephthalic acid of thiophen. In conclusion, the author suggests a mode by which the position of the sulphur in the thiophen group might be definitely determined. It has been shown that ethyl-sulphide is converted into thiophen at an elevated temperature according to the equation:



$$S\left\langle\begin{array}{l}CH_2—CH_3\\ CH_2—CH_3\end{array}\right. = 6H + S\left\langle\begin{array}{l}CH=CH\\ \quad|\\ CH=CH\end{array}\right.$$

Should it be possible to apply the same reaction to propyl and isopropyl sulphide, two thioxylenes should be obtained having the formulæ—

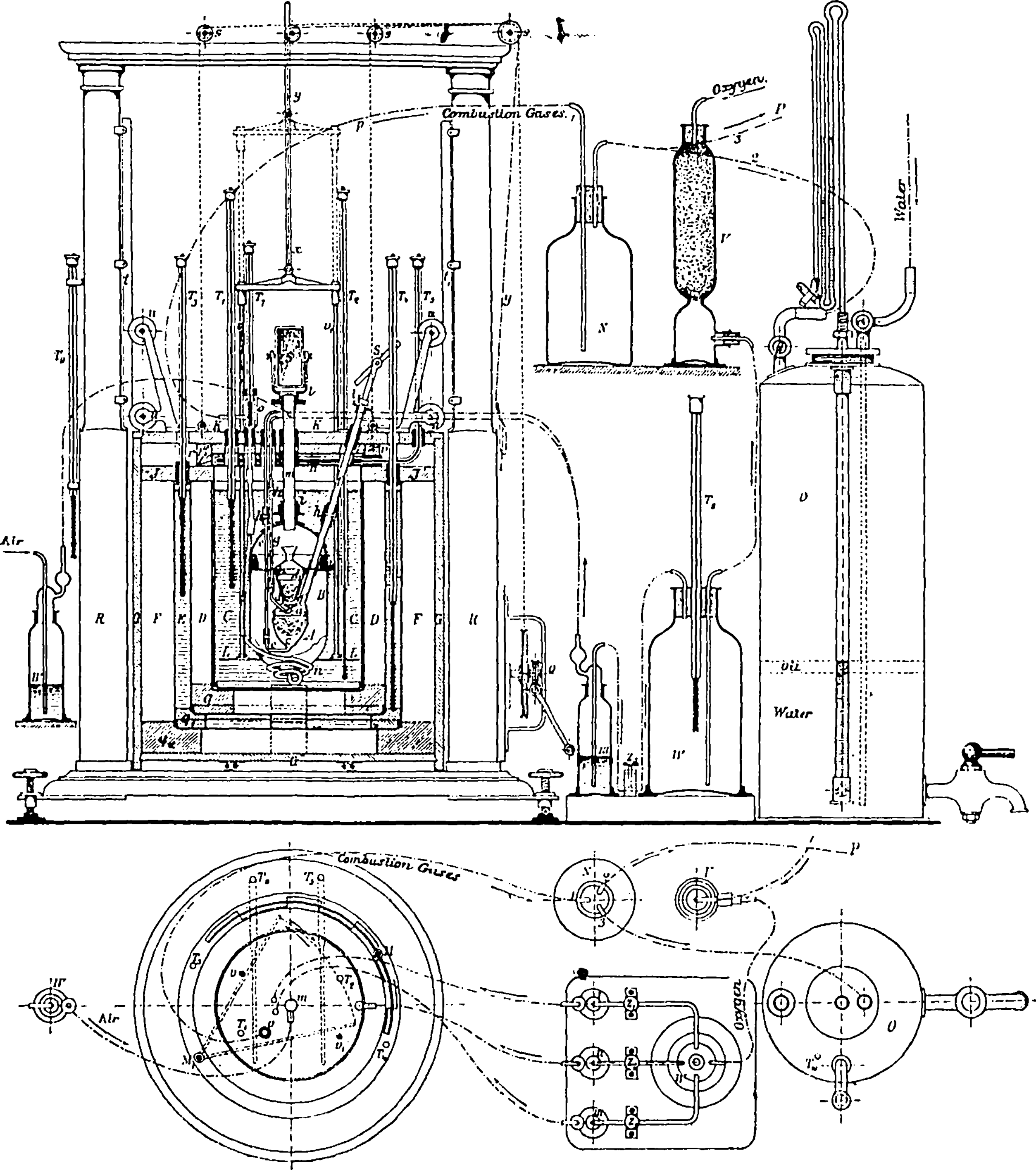


$$S\left\langle\begin{array}{l}CH=C—CH_3\\ \qquad\ |\\ CH=C—CH_3\end{array}\right. \quad \text{and} \quad S\left\langle\begin{array}{l}C(CH_3)=CH\\ \qquad\qquad\ |\\ C(CH_3)=CH\end{array}\right.$$

—D. B.

Nitrothiophens. O. Stadler. Ber. **18**, 530-536.

In a former communication it was shown that on distilling dinitrothiophen melting at 52°, by means of steam, a substance is obtained which melts at a higher temperature. The author has continued his researches in this direction, and finds that the substance has a constant melting point of 78°, and crystallises from water or alcohol in yellow needles. It is an isomeric dinitrothiophen $C_4SH_2(NO_2)_2$. By the action of bromine on dinitrothiophen at an elevated temperature, a substance is obtained which crystallises in white lustrous needles, melting at 112°. It is not a bromine derivative of dinitrothiophen, but represents tetrabromothiophen, the two nitro groups having been removed by the action of bromine at a high temperature. The author has studied the sensitiveness of the colour reaction which dinitrothiophen gives with alcohol and potash, and finds that it is distinctly discernible in a solution containing 0·0000001 grm. of substance. Mononitrothiophensulphonic acid, $NO_2 - C_4SH_2 - SO_3 -$, was prepared by treating three parts of mononitrothiophen with eight parts of fuming sulphuric acid. It gives white hygroscopic crystals, which are resolved into mononitrothiophen when heated. The potassium salt crystallises in grey shining needles, and the calcium salt forms a white crystalline mass.

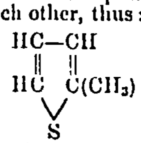

Oxygen.
Combustion Gases.
P
P'
Water
N
T₈
O
Oil
Water
W'
Air
R
G
F
E
D
C
B
A
H
J
K
Q
T₀
T₁
T₂
T₃
T₄
T₅
T₆
S
y
x
m
n
u
v₁
Z₁
Z₂
Z₃
III
W
Combustion Gases
Oxygen
M
Tw

The sulphonic chloride, $NO_2-C_4SH_2-SO_2Cl$, was prepared by heating the potassium salt with phosphorus pentachloride on a water bath. It was converted into the sulphonamide $NO_2-C_4SH_2-SO_2.NH_2$ by treatment with ammonium carbonate. The sulphonamide melts at 172-173°.—D. B.

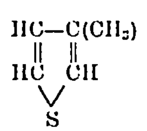
On the Two Isomeric Thiotolens (Methylthiophens).

K. Egli. Ber. **18**, 544

V. Meyer and H. Kreis in 1884 succeeded in separating a methylthiophen from coal-tar toluene, and later on in obtaining another one synthetically by means of thiophen-iodide, methyl-iodide, and sodium. The compounds produced in either way have the same boiling point, and the author investigated the question as to whether they are identical or isomeric. For this purpose the bromine derivatives of both thiotolens were compared with each other, when it was found that their fusing point invariably differed by 12 degrees. They must therefore have a different constitution. This result is quite in accordance with V. Meyer's view on thiophen and its derivatives. In one thiotolen the methyl group and sulphur have the α position, in the second the β position to each other, thus:

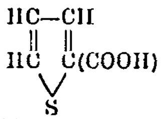


α-thiotolen.

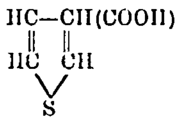


β-thiotolen.

In order to determine the position of the methyl group in both isomeric compounds, they were oxidised with an alkaline permanganate solution. Two different acids were obtained, thus:



α-thiophenic acid.



β-thiophenic acid.

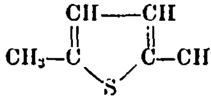
Their bromine substitution product was also prepared. The final result of the investigation is as follows: Thiophen obtained from coal-tar toluene is isomeric, but not identical with that produced synthetically. In the former the methyl group is in the α position, in the latter it is in the β position.—S.H.

Isomeric Thiophensulphonic Acids. J. Langer. Ber. **18**, 553-563.

Dibromothiophenmonosulphonic acid $C_4SHBr_2SO_3H$.—The lead-salt of this acid, formerly described, was converted into the sodium salt, and the latter dried at 120°, and treated with phosphorus pentachloride. The resulting chloride was converted into the amide by treatment with ammonium carbonate. It crystallises in fine needles melting at 146·5-147°. To prepare the *free β-thiophenmonosulphonic acid* $C_4SH_3SO_3H$, the sulphonic chloride described in a previous communication was decomposed by boiling with water, and the liberated hydrochloric acid precipitated with silver hydroxide. The solution was treated with sulphuretted hydrogen, filtered and the filtrate evaporated. A white crystalline mass of strongly acid reaction was obtained. It gave a deep blue coloration when heated with isatin and sulphuric acid. *Barium β-thiophenmonosulphonate* $(C_4SH_3SO_3)_2Ba$ was prepared by saturating the aqueous solution of the free acid with barium carbonate. *Debromination of dibromothiophendisulphonic acid.*—By treating the sodium salt of this acid with sodium amalgam, and adding phosphorus pentachloride to the debrominated substance, the chloride of a new thiophendisulphonic acid was obtained *β-thiophendisulphonic chloride*, $C_4SH_2(SO_2Cl)_2$, crystallises in small plates or laminæ melting at 148-149°. It is readily soluble in ether, has the odour which characterises this class of bodies, and gives a bluish violet solution when heated with isatin and sulphuric acid. *β-thiophendisulphonamide* $C_4SH_2(SO_2NH_2)_2$ was prepared by the action of ammonium carbonate on the sulphonic chloride. It crystallises in white needles melting at about 280°. *Dibromothiophendisulphonic chloride* $C_4SBr_2(SO_2Cl)_2$ was obtained by treating sodium dibromothiophendisulphonate with phosphorus pentachloride. It crystallises from ether in white shining needles, which melt at 215°. *Dibromothiophendisulphonamide* $C_4SBr_2(SO_2NH_2)_2$ was prepared by intimately mixing and heating the sulphonic chloride with solid ammonium carbonate. It is sparingly soluble in hot water, and melts at about 270°. *Lead dibromothiophendisulphonate* $C_4SBr_2(SO_3)_2Pb$ was obtained by converting the anhydride of the acid into the barium salt, decomposing the latter with sulphuric acid, and adding a salt of lead to the filtrate. *Sodium dibromothiophendisulphonate* $C_4SBr_2(SO_3Na)_2+3H_2O$ was prepared by heating the anhydride of dibromothiophendisulphonic acid with soda ley. It crystallises in silky needles which are readily soluble in water. *Ammonium dibromothiophendisulphonate* $C_4SBr_4(SO_3NH_4)_2+H_2O$ was obtained by boiling the anhydride with ammonia. To prepare the *free dibromothiophendisulphonic acid* $C_4SBr_2(SO_3H)_2$ an aqueous solution of the lead salt described in the above was precipitated with sulphuretted hydrogen and filtered. On evaporating the filtrate the free acid was obtained in the form of a solid crystalline white mass readily soluble in water. *Sulphonation of monoiodothiophen.* The author has attempted to prepare a monoiodothiophensulphonic acid, but has not yet obtained successful results. He has, however, deiodated the disulphonic acid by treating its aqueous solution with sodium amalgam. The sulphonic acid was purified by conversion into its chloride, and as the latter contained traces of free iodine it was treated with ammonium carbonate to form the amide. *β-thiophendisulphonamide* $CSH_2(SO_2NH_2)_2$ crystallises in laminæ melting at 142°. It is sparingly soluble in water, and gives a deep blue solution when heated with isatin and concentrated sulphuric acid. *β-thiophenmonosulphonamide* $C_4SH_3SO_2NH_2$ was prepared be converting the barium monosulphonate $(C_4SH_3SO_3)_2Ba$ into the sodium salt, and treating the latter with ammonium carbonate. It crystallises from water in white laminæ which melt at 147°.—D. B.

Thioxen from Coal-tar. J. Messenger. Ber. **18**, 563-568.

Some time ago V. Meyer proved the existence of a thiophen derivative in coal-tar xylene, and gave it the name "thioxen," and the formula—$C_4SH_2(CH_3)_2$. The author has isolated this substance, and investigated its properties. It was prepared from the acid used in the purification of xylene, which contains about 40 per cent. of pure thioxen. The product is a colourless liquid, boiling between 133° and 138°. *Dibromothioxen* $C_4SBr_2(CH_3)_2$, was prepared by adding 50grm. bromine to 50grm. crude thioxen (2mol. Br. to 1mol. thioxen), washing the product with water, boiling it with an alcoholic solution of potash to decompose the bromine addition products formed, then pouring the mass into water, and separating the oil. The latter was distilled with steam, and the distillate fractionated. The first portion boiling between 135° and 165°, consisted mainly of xylene. The second fraction boiling between 205-215°, gave, on redistillation, a liquid boiling at 197-199°. It was composed of a mixture of equal parts of monobromothioxen and monobromothioxen, and could not be separated. The last fraction boiled between 240-250°, and gave a distillate which solidified in the receiver. It was crystallised from alcohol, and yielded long colourless needles, melting at 46°, and boiling between 246-247°, without decomposition. The substance was dibromothioxen. *Octobromothioxen*, $C_4SBr_2(CBr_3)_2$, was obtained from dibromothioxen by treatment with bromine until the latter ceased to react. It crystallises from alcohol in yellow needles, which melt at 114°. *Thioxen* $C_4SH_2(CH_3)_2$. For the isolation of pure thioxen, the method adopted by Meyer and Kreis for separating thiotolen from a mixture of toluene and thiotolen was employed. The pure product is a colourless mobile liquid, of agreeable odour, boiling at 136-137·5°. By the oxidation of thioxen with potassium permanganate, thiophendicarboxylic acid $C_6H_4SO_4$ was obtained. The latter is sparingly soluble in water, but dissolves more readily in ether. Heated to 350°, it does not melt, but sublimes without decomposition. This acid, therefore, appears to be analogous in character to the isophthalic acid in the benzene group, but judging from the melting point of its dimethyl-ether (142°), it is more probably analogous to terephthalic acid. If this be the case, the formula for thioxen would have to be expressed thus—



—D. B.

Fractional Distillation by means of Steam. M. J. Lazarus. Ber. **18**, 577.

THE object was to separate two mixed liquids, one of which was very volatile, whilst the other suffered decomposition at a comparatively low temperature. Naumann has shown that liquids, not miscible with water, when distilled with steam, boil below the temperature at which water boils. The author has, therefore, distilled a number of mixed liquids with steam to ascertain whether they could be fractionated in this manner. The distillation was effected with a moderately powerful pressure of steam, and the distillate collected in two or three fractions. Each fraction was then dried, and fractionated *per se* to ascertain its composition.

EXPERIMENT 1.—25cc. toluene and 25cc. nitrobenzene.

Fraction.	Temperature.	Volume.	Toluene.	Nitrobenzene.
1	90-95°	21cc.	19cc.	——
2	95-98°	6cc.	3·5cc.	——
3	98°	23cc.	——	23cc.

Recovered: 22·5cc. toluene and 23cc. nitrobenzene.

EXPERIMENT 2.—25cc. benzene and 25cc. nitrobenzene.

Fraction.	Temperature.	Volume.	Benzene.	Nitrobenzene.
1	96°	23·5cc.	22·00cc.	——
2	96-98°	4·0cc.	1·75cc.	——
3	98°	22·5cc.	——	22·5cc.

Recovered: 23·75cc. benzene and 23·5cc. nitrobenzene.

EXPERIMENT 3.—30cc. nitrobenzene and 5cc. toluene.

Fraction.	Temperature.	Volume.	Toluene.	Nitrobenzene.
1	95-98°	6cc.	4·25cc.	——
2	98°	29cc.	——	29cc.

Recovered: 4·25cc. toluene and 29cc. nitrobenzene.

EXPERIMENT 4.—25cc. toluene and 25cc. xylene.

Fraction.	Temperature.	Volume.	Toluene.	Xylene.
1	84-91°	22cc.	16·5cc.	2cc.
2	91-98°	28cc.	3·0cc.	18cc.

Recovered: 19·5cc. toluene and 20cc. xylene.

EXPERIMENT 5.—25cc. benzene and 25cc. carbon-disulphide.

Fraction.	Temperature.	Volume.	Carbon-disulphide	Benzene.
1	64°	22	19cc.	——
2	64-67°	6	3cc.	——
3	67°	22	——	21·5cc.

Recovered: 22cc. carbon-disulphide and 21·5cc. benzene.

EXPERIMENT 6.—25cc. oil of turpentine and 25cc. nitrobenzene.

Fraction.	Temperature.	Volume.	Oil of Turpentine.	Nitrobenzene.
1	95-97°	21cc.	19cc.	——
2	97-98°	4cc.	2cc.	——
3	98°	25cc.	——	24cc.

EXPERIMENT 7.—25cc. aniline and 25cc. nitrobenzene.

The mixture distilled at 98°, and was collected in two fractions, each measuring 25cc. The first contained 15cc. aniline and 10cc. nitrobenzene, and the second 10cc. aniline and 15cc. nitrobenzene. Benzene and toluene could not be separated perfectly, even when large quantities were operated on. The author has used this process with success for the separation of iodothiotolen and toluene and iodothioxen and xylene, an operation which has hitherto been attended with great difficulties and much loss, owing to the decomposition of the iodine compounds at comparatively low temperatures.

—D. B.

Occurrence of Carboxylic Acids in Coal-tar. K. E. Schulze. Ber. **18**, 615.

HITHERTO bodies of the phenol group only have been shown to exist in the oxygenated constituents of coal-tar, of which phenol and α- and β-naphthol have been isolated, whilst the presence of cresols, xylenols, etc., has been inferred from isolated derivatives of these compounds. In pursuing his investigations on the high-boiling phenols of coal-tar, the author discovered a carboxylic acid, which was found to be benzoic acid. It was isolated in the following manner:—In order to obtain cresols and xylenols, the residues from the distillation of phenol were subjected to fractional distillation. The last portions came over between 235° and 240°, and from this boiling point the author inferred the presence of pseudocumenols, of which there are three isomers. To separate the latter, the oil was treated with soda ley, and distilled with steam. The residue contained the sodium salt of the strongest acid present in the mixture. On adding sulphuric acid, a brown-coloured oil was liberated, which, on cooling, solidified in the form of needles, melting at 123°, and boiling at 249·2°. The silver salt was prepared, and its formula agreed closely with that of silver benzoate, $C_7H_5O_2Ag$. 100grm. of crude material gave 8grm. of recrystallised benzoic acid. As to the formation of this acid, the author states that it cannot be produced by any of the processes in vogue for preparing carbolic acid; he is, moreover, of opinion that it is contained in the products of the distillation of coal-tar, and passes into the carbolic acid during the treatment of the oil with soda ley. —D. B.

Thiophen from Erythrol. C. Paul and J. Tafel. Ber. **18**, 688.

IT has recently been demonstrated that the group CHOH - CHOH - CHOH - CHOH, which is contained in pyromucic acid, passes into the thiophen ring on treatment with sulphuretted hydrogen. The simplest body in which this complex of atoms is found is erythrol, $CH_2OH - CHOH - CHOH - CH_2OH$, which on heating with phosphorus pentasulphide gave thiophen.—D. B.

Remarkable Formation of Anthracene. H. Köhler. Ber. **18**, 859.

IN a communication on high-boiling phenols contained in coal-tar, Schulze expressed the opinion that the phenols are the primary products of the destructive distillation of coal. By continuing the action of heat they are partly split up, water being liberated and high-boiling hydrocarbons formed, whilst another portion is reduced to low-boiling hydrocarbons, or is eventually converted into coal-gas through the complete disintegration of the molecules. A portion resists decomposition by heat at the point at which the various reactions reach an equilibrium. Schulze arrived at this theory by the observation that the phenols separated from anthracene oil when subjected to distillation yield liquid hydrocarbons with partial liberation of water. Some time ago the author made an observation which appears to confirm to some extent Schulze's view. In working up the high-boiling portions of crude carbolic acid, a cast-iron still was charged with "carbolic residues," which distilled above 205°. They were free from hydrocarbons, and gave a clear solution with caustic soda. On distillation about two-thirds passed over in the ordinary way as a pale yellow-coloured oil, having the odour peculiar to phenol. After this the thermometer rose rapidly above 300°, and from the condenser dense white fumes issued, whilst the distillate in the receiver assumed a consistence like that of butter. On opening the still it was found to be half-filled with a spongy mass of coke. The distillate was washed with alkali to remove the liquid constituents (phenols), and was tested for anthracene by Luck's method, which gave thirty-five per cent. of pure anthracene. The presence of methylanthracene, phenanthrene, etc., in the crude product was established by its properties. The author then made a number of distillations in the same boiler, but failed to obtain anthracene. He is of opinion that the decomposition was caused by local superheating in the still. The probability of this assumption is strengthened by the fact that the crude carbolic acid in question had been obtained from light oil, which contains phenols of such complicated constitution that it would be difficult to conceive the formation of anthracene therefrom by the mere liberation of water; moreover, the whole course of the reaction—production of large quantities of non-condensible substances and separation of coke—points to a more complex decomposition. Be this as it may, the fact can no longer be doubted that under suitable conditions anthracene can be formed from phenolic bodies extracted from the light-boiling portions of coal-tar.

Many chemists are of opinion that the greater part of the anthracene obtainable from coal-tar is produced during the process of distillation, whilst it has been found that the yield of anthracene varies according as the fire is allowed to play round the whole or only part of the still. This view appears to be supported by the author's observations.—D. B.

Synthetical Lutidine. W. Epstein. Ber. **18**, 883.

IN consequence of a recent communication made by Haitinger concerning the production of a lutidine melting at 147-151° from derivatives of dehydracetic acid, the author states that in investigating the derivatives of a body obtained by the aid of the condensation of acetoacetic ether with aldehyde ammonias, according to Hantzsch's method, he obtained a lutidine melting at 145-146°. It is different in properties to orthoparadimethylpyridine (m.pt. 154-155°), first prepared by Hantzsch from lutidinetricarboxylic acid, and subsequently in larger quantities from pseudolutidostyril. It differs also from Ladenburg and Roth's lutidine (m.pt. 142-143°), found in commercial picoline. The platinochloride of the author's lutidine melts at 216°, the aurochloride at 119°, the picrate at 161°, the dichromate at 92°, and the mercuro-chloride at 155°.—D. B.

Caucasian Petroleum. P. Milkowsky. J. Russ. Phys. Chem. Soc. 1885 [2], 37.

THE author has subjected the portion of Bibi-Eibat and Balachany petroleum boiling between 85° and 105° to a succession of fractional distillations over sodium, and obtained the following products:—

	90-91°	93-94°	96-97°	100-101°
Sp. gr. at $\frac{0^\circ}{0^\circ}$ ..	0·7170	0·7521	0·7650	0·7778
" " $\frac{17·5^\circ}{0^\circ}$ ..	0·7320	0·7390	0·7515	0·7621
Vapour density	3·65	3·55	3·57	3·57

The fraction boiling between 100° and 101° was heptanaphthene, C_7H_{14}. By passing chlorine through this hydrocarbon at the ordinary temperature, 72·8 per cent. of chlorides lighter than water, and 27·2 per cent. of chlorides heavier than water, were produced, whilst at an elevated temperature 58 per cent. of heavy, and 42 per cent. of light, chlorinated derivatives were formed. Bromine acts readily on heptanaphthene, whilst iodine dissolves therein without further action.—D. B.

Examination of Caucasian Petroleum. W. Markownikow. J. Russ. Phys. Chem. Soc. 1884 [2], 294.

OCTONAPHTHENE.—This hydrocarbon has already been isolated from Balachany petroleum, and the author has recently extracted it from the petroleum which occurs at Bibi-Eibat. It boils at 119°, has a sp. gr. of 0·7649 at $\frac{0^\circ}{0^\circ}$, and 0·7503 at $\frac{18^\circ}{0^\circ}$, and is composed of 85·3 per cent. of carbon, and 14·7 per cent. of hydrogen. When chlorinated according to Schorlemmer's method, a number of isomeric chlorides are obtained. The chloride boiling at 174-176° was heated to 60° with dry calcium iodide, and the resulting iodide converted into the ether of the octonaphthenealcohol by treatment with silver acetate. The ether boils at 196-200°. The iodide prepared from the chloride boiling between 169-172° was converted into octonaphthylene, $C_{18}H_{14}$, by treatment with moist oxide of silver. It boils at 118-121°, and gives a liquid brominated derivative, $C_{18}H_{14}Br_2$. M. Putochin has obtained iso-octonaphthene from the intermediate products formed in the isolation of octonaphthene and nononaphthene by repeated fractionation over sodium, and treatment with fuming sulphuric acid. It boils at 122-124°, and has the formula $C_{18}H_{16}$. Its sp. gr. is 0·7768 and 0·7765 at $\frac{0^\circ}{0^\circ}$, and 0·7637 at $\frac{17·5^\circ}{0^\circ}$. The chlorinated derivatives have higher boiling points than the corresponding derivatives of octonaphthene. *In order to obtain iso-octonaphthylene* boiling at 128-129°, the chlorinated derivative is treated with an alcoholic solution of potash.—D. B.

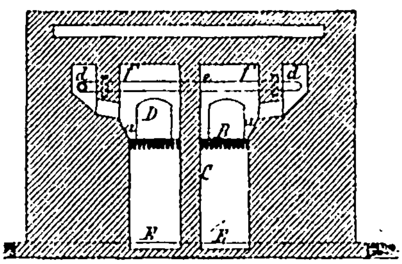
Anthracene in Tar from Water-gas. Arthur H. Elliott. Amer. Chem. J. **6**, 248-252.

IN the fraction of water-gas tar boiling at 210-230° (product of the destructive distillation of light petroleum naphtha), 5·8 per cent. of anthracene was found, and 2·08 per cent. in the higher boiling fractions. The total yield from the original tar amounted to 2·6-2·9 per cent.
—J. B. C.

Improved Apparatus for Carbonising Bones and Turf, and for Coking and Distilling Brown Coal, Pit Coal, and other Carbonaceous Materials by means of Superheated Steam. B. J. B. Mills, London. From A. Zwillinger, Vienna. Eng. Pat. 6995, April 29, 1884.

THE apparatus employed for carrying out this invention is principally composed of a furnace A (Figs. 1, 2 and 3), for superheated steam, several cast-iron retorts communicating with a common condenser, and of a gasometer of the usual form. The fireplace of the furnace is divided into two chambers, so that it is heated by two separate fires, and along both sides of the flue conduits are formed in the brickwork, communicating with the flue at certain intervals by means of small apertures. The steam pipes *c* rest in two vertical walls, so that the elbow joints *d* are placed right and left in narrow channels f_1, which are in connection with the furnace proper by narrow flues f_2. The steam enters at *g*, and after traversing the heated pipes *c*, it passes through pipes *h* and branches h_1, which are provided with many small holes, *into cast-iron cylinders G, containing the material* to be treated. The fire-gases travel through a flue *k* into an annular space *j*, heating thus the steam pipes and the cylinders G, and escape at *l* into the chimney. The vapours and gases evolved *in G are conveyed to a* condenser H, and thence through *n* to coolers. The

Fig. 1.



admission of air to the fire-places can be carefully regulated by means of slides in the ash-pit doors c, and in order to prevent the destruction of the cast-iron pipes c and cylinders G by oxidation, they are covered with a fire-proof material, consisting of 120 parts fire-clay, 40 parts animal charcoal and 2 parts cow-hair.—S. H.

Fig. 2.

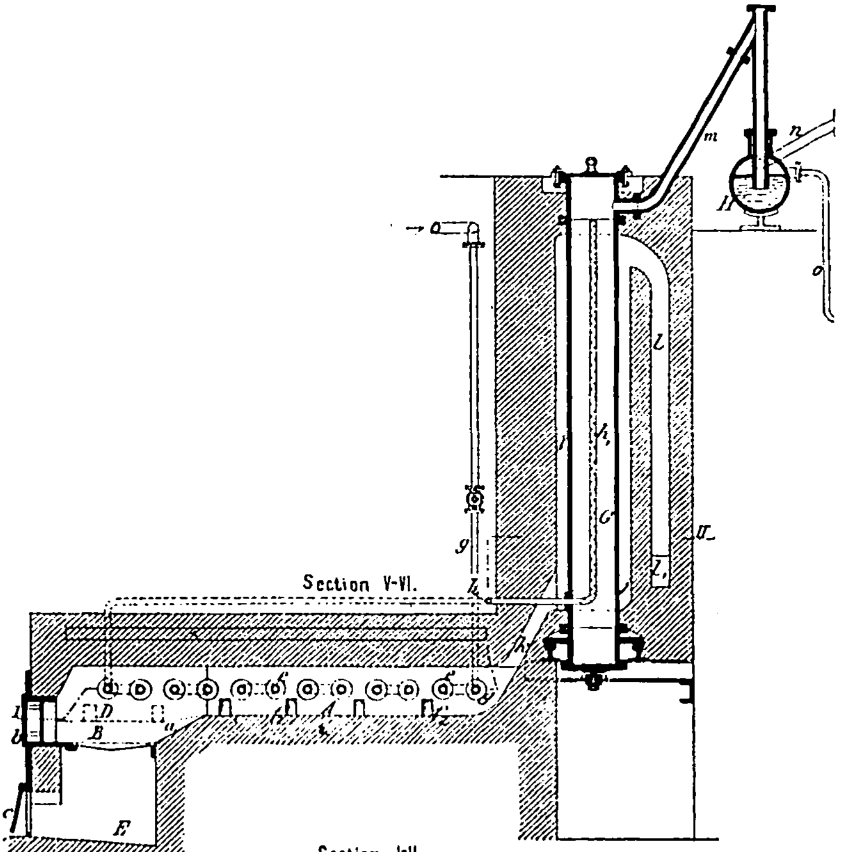
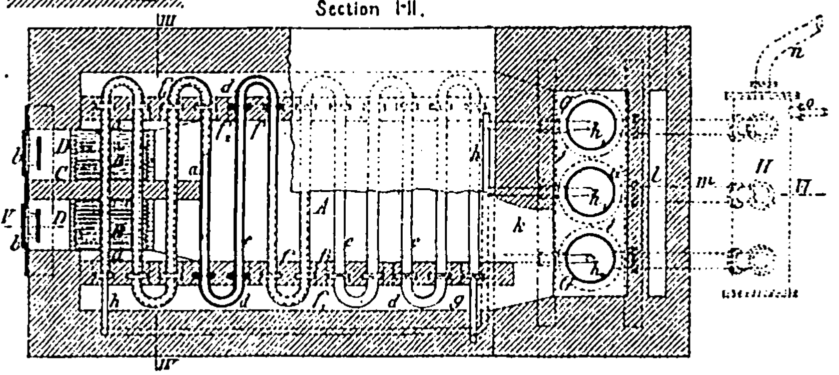


Fig. 3.



Improvements in Apparatus for Distilling Shales and the Like. R. Bell, Broxburn, Scotland. Eng. Pat. 9285, June 21, 1884.

The inventor proposes to build a furnace, erect over the same the lower chamber of the retort, and mount over the latter the upper chamber. Surrounding these chambers is an outer wall, the intervening space being in communication with the furnace by suitable openings. In the lower part of the bottom chamber there is a shoot which passes in a downward direction at the side of the outer wall, and opens into the furnace beneath, and through it the contents of the bottom chamber of the retort are discharged into the furnace to further utilise their heat. Between the chambers of the retort a valve or slide is arranged, which can be operated from the outside of the apparatus to allow the contents of the upper chamber to pass into the chamber below as required. The retort is provided with openings and pipes for the outlet of the volatile products of distillation at different levels.—D. B.

Improvements in the Treatment of Oils and Tars obtained from Coke-Ovens, for the Production of Valuable Hydrocarbons. C. E. Bell, Durham. Eng. Pat. 9510, June 27, 1884.

At the present time oils and tars are obtained from coke ovens which do not contain in their natural state any considerable proportion of benzene, naphthalene, anthracene, etc. By passing these oils in the state of vapour through retorts charged with coke heated to redness, the inventor claims to obtain large quantities of hydrocarbons of the above series.—D. B.

Improvements in the Treatment of the Gases from Coke Ovens for the Recovery of Bye-products therefrom, and in Apparatus for that Purpose. H. Simon, Manchester. From F. Carvés, St-Etienne, France. Eng. Pat. 15,920, December 3, 1884.

This invention relates to coke ovens, more particularly those known as the Simon-Carvés coke ovens, wherein the combustible gases generated in the coking chambers are first treated for the separation of tar and ammoniacal liquor, and are then used as gaseous fuel. According to the present invention, the gases before being burned are subjected to a further treatment, whereby the inventor separates from them the light hydrocarbons which are still retained in a gaseous form therein. For this purpose, the gases, after purification from ammoniacal and other easily condensible constituents, are subjected to the intimate action of heavy oils capable of absorbing the light hydrocarbons, the oils being then heated or otherwise acted on so as to separate from them the light hydrocarbons. The various parts of the apparatus for effecting the separation of the light hydrocarbons are described and illustrated in the original.—D. B.

An Improved Process of Manufacturing Benzene and its Homologues by the Distillation of Coal, Wood, Turf, Oil and other similar Substances. N. A. Hélouis, Paris. Eng. Pat. 6904, April 28, 1885.

This patent is similar in principle to a former invention, for which the author obtained provisional protection only. (See this Journal, vol. iii. p. 438).—D. B.

IV.—COLOURING MATTERS AND DYES.

*Process for the Production of Indigo.** Dingl. Polyt. J. **255**, 356.

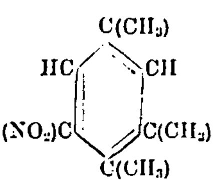
According to H. Müller (Ger. Pat. 30,329, June 12, 1883), benzaldehyde is chlorinated in presence of a dehydrating agent, and meta-chlorbenzaldehyde obtained as a colourless liquid of 1·246 relative density at 15°, and boiling at 206°. By the nitration of this compound, meta-chlor-orthonitrobenzaldehyde is formed and may be precipitated from the nitrating mixture with ice-cold water; crystallised repeatedly from alcohol, it forms yellowish needles, melting at 60°. By dissolving metachlor-orthonitrobenzaldehyde in acetone, and adding to this solution first a little water, then dilute soda-lye, a formation and separation of chlorindigo takes place in a short time. The chlorindigo thus prepared closely resembles pure indigo; it is a deep blue crystalline powder with coppery lustre, without taste or smell, insoluble in water, alcohol, and ether, dilute acids, and alkalies, somewhat soluble in hot chloroform, aniline, and benzalchloride. When heated, chlorindigo sublimes with partial decomposition; distilled with sodium hydrate, it breaks up into carbonic acid and chloraniline. Treated with concentrated sulphuric acid, it forms a sulphonic acid soluble in water, which dyes wool blue. Treated with reducing agents, chorindigo yields a vat which may be employed in the same manner as that from ordinary indigo. Chlorindigo exposed to the action of nascent hydrogen is converted into indigo.—W. D. B.

Azo-dyes. Soc. Anon. de Matières Colorantes de St-Denis. Dingl. Polyt. J. **255**, 404.

Azo-dyes which impart a pure yellow to vegetable fibres capable of resisting a soap bath of 60°, are obtained (Ger. Pat. 29,991, March 25, 1884) by the action of para- or meta-diazobenzoic acid on diphenylamine or monobenzylaniline. For example, 5kilos. sodium para- or meta-amidobenzoate are dissolved in 200 litres of water, and 6·6kilos. of a 33 per cent. solution of sodium nitrite are added. The mixture is acidified with 9·4 kilos. of sulphuric acid of 53° B. previously diluted with water; and care is taken, by the addition of ice, to keep the temperature below 20°. This mixture is then poured upon 5·3kilos. diphenylamine or monobenzylaniline, which is suspended in 170 litres of acidified water. After four to eight days the reaction is complete. The insoluble dye is filtered, washed and dried. The dye produced by the meta-compound is yellower than that produced by the para-compound; the latter approximates more to orange yellow. To apply this dye to cotton goods, the dye is thickened with starch or tragacanth, and then with the necessary quantity of alkali to convert the acid dye into a salt. On boiling, cooling and adding excess of acetic acid, the insoluble acid dye is again set free, but in a condition suitable for fixing on the goods. Chromium acetate serves best as mordant. After printing the goods are steamed. The dye retains its brightness in a soap bath heated to 60°. To obtain a bright yellow after simple washing with water, an aluminium salt is used as mordant. The yellow dye obtained with chromium mordant combines very well with artificial alizarin red. —J. T.

Nitropseudocumene, Pseudocumidine and Pseudocuminol (1, 3, 4, 5). E. Edler. Ber. **18**, 629.

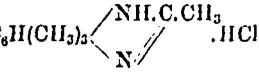
The derivatives of pseudocumene known up to the present have the symmetrical position 1, 3, 4, 6. The author prepared the isomeric compounds, whose constitutional formula may be represented by the nitropseudocumene, thus—



With this object in view, the common pseudocumidine was first converted into the acetyl and thence into the nitro compound. After separating the acetyl group and treating the resulting nitropseudocumidine with nitrous acid and alcohol, a nitropseudocumene was obtained. In order to determine its constitution, the nitroacetylpseudocumidine

$$C_6H(\overset{1}{C}H_3)(\overset{3}{C}H_3)(\overset{4}{C}H_3)(\overset{5}{N}O_2)(\overset{6}{N}H)(C_2H_3O)$$

was reduced, whereby a base was produced whose hydrochloride had the following composition—



$$C_6H(CH_3)_3\left\langle\begin{matrix}NH.C.CH_3\\ N\end{matrix}\right. .HCl.$$

The author concludes that as it was found impossible to split off the acetyl group with alcoholic potassium hydrate, the nitro and amido group in nitropseudocumidine must be in the ortho position to each other. This conclusion proved correct, when by reducing nitropseudocumidine a base pseudocumylenediamine,

$$C_6H(CH_3)_3(NH_2)_2$$

was obtained, which had all the characteristic properties of an ortho-diamine, as stated by Ladenburg (Ber. xi. 600 and 1650).—S. H.

Preparation of Nitrosonaphthols. R. Henriques and M. Ilinski. Ber. **18**, 704.

β-Naphthol: 1 part commercial β-naphthol and 0·75 part zinc chloride are dissolved in 6 parts alcohol; 0·5 part sodium nitrite dissolved in little water is then added to the boiling alcoholic solution. The liquid turns red, and whilst the boiling is continued the red brown zinc salt separates out. On standing at rest over night, the salt is filtered and washed with alcohol. It is then suspended in 10 parts water and digested for a short time with 0·8·1 part caustic soda until it has been converted into the green crystallised sodium salt. The latter is filtered, washed with water and decomposed at the common temperature with hydrochloric acid. The nitrosonaphthol thus obtained, after filtering and washing, is almost chemically pure, and needs no further purification for the use as reagent. The yield is 110% to 115% of the β-naphthol used.

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* See also this Journal, iii. 111.

α-naphthol: A similar treatment of the α-naphthol for obtaining nitroso-α-naphthol is not advisable, the α-naphthol not being easily attacked by nitrites of metals. In order to produce the α-derivate in an almost pure state, a solution of 1 part commercial α-naphthol and 1 part zinc chloride in 6 parts alcohol is heated to the boil. A concentrated aqueous solution of 0·5 part sodium nitrite is added, and the boiling continued for two to three hours. After standing over night, the red zinc salt of β-nitroso-α-naphthol separates out, which is, however, permeated with fine yellow needles of α-nitroso-α-naphthol, whose zinc salt is decomposed by boiling with alcohol. The mass is filtered, washed with cold alcohol, and the two nitrosonaphthols are separated with an alcoholic potassium hydrate solution (Ber. xvii. 2590) without previously decomposing the zinc salt with hydrochloric acid. The alcoholic filtrate contains undecomposed α-naphthol and α-nitroso-α-naphthol. A large quantity of water is added, when only the nitroso compound separates out, whereas α-naphthol does not crystallise out but after some time. The precipitate is filtered immediately and washed with water. The nitrosonaphthol, thus precipitated, is nearly as pure as that previously prepared from the residual mixture. If it contains some impurities or traces of undecomposed α-naphthol it is washed with benzene. 100grm. naphthol yielded 50gr. yellow (β) and 40gr. white (α) nitrosonaphthol.—S. H.

Colouring Matters from Lepidine. S. Hoogewerff and W. A. van Dorp. Rec. Trav. Chem. **3**, 337-362.

The present paper is a continuation of the authors' previous researches, and also the results of their investigations on cyanine and on a new colouring matter first obtained by Spalteholz.

Dimethylcyanine-chloride is obtained from the corresponding iodide by the action of AgCl. Diethylcyanine-bromide can be prepared in the same manner as the iodide from diethylquinolinebromide and ethyllepidinebromide, and dissolves in alcohol with a pure blue colour, which is not fast to light or carbonic acid. By the action of potash upon the iodides of methyl-*p*-toluquinoline and methylquinoline, a resin is formed, soluble in alcohol with red colour. If a mixture of 2 parts of methyl-*p*-toluquinoline iodide with 1 part of methyllepidine iodide, in aqueous solution, be heated with potash equivalent to one-half of the iodine present, dimethylmethylcyanineiodide of the formula

$$C_{22}H_{21}N_2I + H_2O$$

is obtained, crystallising in blue needles. The authors find that the colouring matter of Spalteholz has the formula $C_{23}H_{23}N_2I$, and not $C_{23}H_{25}N_2I$, the reaction taking place according to the equation

$$C_9H_7N_1C_2H_5I + C_{10}H_9N_1C_2H_5I = C_{23}H_{23}N_2I + 2H + HI.$$

This substance, isomeric, with diethylcyanine, is prepared from quinaldine-ethyliodide with 1·9 part quinoline-ethyliodide in 25 parts of alcohol, and the calculated quantity of potash gradually added, the mixture being heated on the water-bath. On distilling off a portion of the alcohol, the new compound crystallises out. A crystalline body can also be prepared in a similar way from ethylquinaldine alone.

Dimethylcyanine-iodide.—The two representatives of the colouring matter known as cyanine have been shown by the authors to be mixtures, the composition of the pure compound being expressed by the formula $C_{29}H_{35}N_2I$, formed according to the equation

$$C_9H_7N_1C_5H_{11}I + C_{10}H_9N_1C_5H_{11}I = C_{29}H_{35}N_2I + H_2 + HI.$$

The best yield is obtained by digesting amyliodide in alcoholic solution, with the corresponding bases—quinoline and lepidine—on the water-bath, the requisite amount of potash being added. On distilling off a portion of the alcohol, the colouring matter may be extracted from the residual resin by means of acetone. By heating amyl-lepidine iodide with an alkali, a red substance is formed, soluble in alcohol; but it has not been crystallised.—J. B. C.

Manufacture of Colouring Matter. Charles Denton Abel, London. From the Farbwerke vormals Meister Lucius and Brüning of Höchst-am-Main, Germany. Eng. Pat. 6311, April 12, 1884.

The object of this invention is to prepare violet and blue colouring matters of the rosaniline series by the action of perchlormethyl-formate upon tertiary aromatic amines in the presence of condensing agents. As a typical example, the inventors give the following method of preparing hexamethyl-violet:—25lb. of dimethylaniline are mixed with 5½lb. of perchlormethyl-formate, and 12lb. of anhydrous zinc chloride gradually added to the mixture. The violet gradually forms as a crystalline mass, which is freed from excess of zinc chloride by washing with water, and then purified in the usual way.—R. M.

A Process or Processes for Producing β-naphtholdisulphonic Acids, Separating the same and Obtaining Colouring Matters therefrom. Jasper Wetter, London. From the Leipziger Anilinfabrik, Beyer and Kegel, of Lindenau-Leipzig, Saxony. Eng. Pat. 7097, May 1, 1884.

Four parts of sulphuric acid of 66° Bé. are heated to 125-135° C., and one part β-naphthol is then quickly introduced, the temperature mentioned being maintained for four to five hours. The product is dissolved in three times its weight of water, caustic soda equal to half the weight of the acid employed is added, and the solution is then saturated with common salt. On cooling, a portion of the acid salts of the sulpho acids crystallises out, and the remainder is left in the solution. The crystalline portion is filtered off and washed with brine, and both this and the soluble portion may be directly employed for the production of azo colours, the latter giving the more yellow shades. The inventors give a list of the diazotised bases employed, and the colours obtained respectively from the soluble and the crystalline modifications of the disulpho acids.—R. M.

A Process or Processes for Separating the Different β-naphtholmonosulphonic Acids from a Mixture of the same, and for Obtaining Colouring Matters therefrom. Jasper Wetter, London. From the Leipziger Anilinfabrik, Beyer and Kegel, of Lindenau-Leipzig, Saxony. Eng. Pat. 7098, May 1, 1884.

According to this invention, β-naphthol when heated to 100° C. with the quantity of strong sulphuric acid, theoretically necessary to form a monosulphonic acid according to the process of H. E. Armstrong, gives rise to a mixture of two isomeric monosulphonic acids, which can be readily separated by treating the solution of the free acid or the sodium salt with common salt. Both the portion which separates out, and the soluble portion, can be directly employed for the preparation of azo colours.
—R. M.

Estimation of Orthotoluidine and Paratoluidine, if present together in Mixtures, by means of the Specific Gravity. G. Lunge. Chem. Ind. **8**, 74.

The mixture of ortho- and paratoluidine forms a liquid at 15° C. as long as it does not contain more than 50 per cent. of paratoluidine. It is, therefore, easy to determine its specific gravity at 15° C. If it contains 60 per cent. of paratoluidine, the temperature at which the specific gravity is taken must be raised to 20° C. Beyond this percentage the author does not think his method available, owing to the different mixtures showing only very small differences in their specific gravities, and it being well-known that observations taken at temperatures artificially raised lose much in accuracy. For precise purposes the specific gravity must be found by means of a sensitive balance and picnometer, but for practical purposes the use of an accurate hydrometer is admissible. Absolutely indispensable is an accurate observance of the temperature, 1° C. more or less causing a difference of 7 per cent. Furthermore, the mixture must not contain water or aniline or xylidine, and if so the former must be removed by means of fused potassium hydrate and successive distillation. If the temperature of observation is not exactly 15° C., the specific gravity found, must be adjusted for each ± 1° C.; if it be more than 1·0008 it must be corrected by ∓ 0·0008; if it be less than 1·0008 by ∓ 0·0007.

TABLE OF THE PERCENTAGE OF MIXTURES OF ORTHO- AND PARATOLUIDINE.

Specific Gravity Taken—

(a) at 15° C., and based upon water at 15° C.

Specific Gravity.	Orthotoluidine per cent.	Specific Gravity.	Orthotoluidine per cent.	Specific Gravity.	Orthotoluidine per cent.	Specific Gravity.	Orthotoluidine per cent.	Specific Gravity.	Orthotoluidine per cent.
1·0037	100	1·0024	88½	1·0011	78½	0·9998	68	0·9985	58
1·0036	99	1·0023	88	1·0010	77½	0·9997	67	0·9984	57½
1·0035	98	1·0022	87	1·0009	77	0·9996	66½	0·9983	56½
1·0034	97	1·0021	86½	1·0008	76	0·9995	65½	0·9982	56
1·0033	96	1·0020	86	1·0007	75	0·9994	65	0·9981	55
1·0032	95	1·0019	85	1·0006	74	0·9993	64	0·9980	54½
1·0031	94	1·0018	84½	1·0005	73	0·9992	63	0·9979	54
1·0030	93½	1·0017	83½	1·0004	72½	0·9991	62	0·9978	53
1·0029	92½	1·0016	82½	1·0003	72	0 9990	61½	0·9977	52½
1·0028	91½	1·0015	82	1·0002	71	0·9989	61	0·9976	51½
1·0027	91	1·0014	81	1·0001	70	0·9988	60	0·9975	51
1·0026	90	1·0013	80	1·000	69	0·9987	59	0·9974	50
1·0025	89½	1·0012	79½	0·9999	68½	0·9986	58½	...	...

(b) at 20° C. and based upon water at 15° C.

Specific Gravity.	Orthotoluidine per cent.	Specific Gravity.	Orthotoluidine per cent.	Specific Gravity.	Orthotoluidine per cent.	Specific Gravity.	Orthotoluidine per cent.	Specific Gravity.	Orthotoluidine per cent.
0·9930	50	0·9936	48	0·9933	46	0·9930	44	0·9927	41
0·9938	49½	0·9935	47½	0·9932	45	0·9920	43	0·9926	40
0·9937	48½	0·9934	46½	0·9931	44½	0·9928	42	...	...

—S. H.

V.—TEXTILES : COTTON, WOOL, SILK, Etc.

The Influence of Certain Processes in the Manufacture of Cotton Fabrics upon their Strength. Dingl. Polyt. J. 255, 349.

Scheurer (*Bulletin de Mulhouse*, 1883, p. 76) finds that water at a temperature of 150° has no marked tendering effect upon cotton, even when the action is continued for eight hours. At temperatures above 160° the tendering action of water is well-marked and considerable. Maintained in contact with air for one hour at a temperature of 170°, the fabric does not appear to suffer any diminution of strength. Grosseteste (*Ibid.* p. 65) contributes some experiments as to the temperatures to which fabrics attain during singeing. The fabric was saturated with a solution of an aniline salt, potassium ferricyanide and potassium chlorate, which at a temperature of 80° developed a green, at 100° a black, and between 120° and 130° a reddish-brown colour. With Tulpin's singeing machine the fabric became green; by passing three times through Blanche's apparatus with three flames, a uniform black was developed.

An extended series of experiments by Haebler upon the influence of the bleaching processes upon the strength of cotton fabrics is published in the *Mittheil. des Technologischen Gewerbe-Museums*, Section für Färberei, Vienna, 1884, p. 11. These experiments show that any tendering of a fabric cannot be ascribed to the action of chlorine or to any part of a properly conducted bleaching process. The following numbers, expressing the breaking weight in kilogrammes of a fabric after different stages of the bleaching process, may be adduced in proof of the foregoing statement :—

Stage of Process.	Breaking Load. Kilos.
Unsized	4·656
Sized	5·029
Singed	5·206
Boiled in lime, washed	4·694
Acidified with HCl, washed	4·664
Boiled in soda, washed	4·799
Treated with chlorine, washed	5·204
Acidified with H_2SO_4, washed	5·345
Dressed	6·423

A second series of experiments is devoted to the question of the tendering action of various mordants as follows :—

No. 1. Aluminium aceto-sulphate

$$Al_2(SO_4)_2(C_2H_3O_2)_2$$

with sodium phosphate.

No. 2. Sodium aluminate $Al_4Na_6O_9$ with ammonium chloride.

No. 3. Aluminium sulphocyanide $Al_2(CyS)_6$ with sodium phosphate.

No. 4. Pure acetate of alumina $Al_2(C_2H_3O_2)_6$.

No. 5. 140g. $FeSO_4$, 0·5l. H_2O, 55g. sodium acetate.

No. 6. Same as No. 5, but 110g. sodium acetate.

No. 7. Bistre: 100g. crystallised manganous chloride, 0·5l. of water, 55g. sodium acetate.

No. 8. Chromate of lead dye.

No. 9. Same as No. 8, but subsequently treated with lime water.

The breaking loads of portions of the same fabric treated with the above are here given:—

No. of Mordant.	Breaking Load. Kilos.
0	5·481
1	5·371
2	5·269
3	5·368
4	4·920
5	4·230
6	4·495
7	5·199
8	4·620
9	4·776

The conclusion to be drawn from the figures adduced is that cotton fabrics are weakened by the application of the mordants and compounds detailed above.—W. D. B.

VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

Recent Progress in the Chemistry of Calico Printing.

A Lecture delivered before the Verein z. Bef. der Gewerbefleisses, Berlin. O. N. Witt.

After a general introduction, in which the author discusses the many-sided qualifications necessary to the discharge of the functions of a textile colourist, he proceeds to the treatment of the more important items in the recent progress of the art, viewed from the chemical standpoint. The processes of continuous steaming, and more particularly the apparatus invented by Mather & Platt, for instantaneous steaming, are exerting considerable influence upon the methods of development and fixation. The latter is of especial advantage in the development of aniline black, in the removal of the acetic acid present in the colour mixtures, as a preliminary to the longer exposure to steam in the ordinary apparatus, and in which the presence of the acid is objectionable, also in the fixation of many mordants, such as alkaline chrome. The author then refers to recent advances in the chemistry of cellulose, more especially to the researches of Witz and of Cross and Bevan, and the modifications of the fibre substance under oxidising influences, which they have brought to light (see this Journal, iii. 206 and 291). The power which cellulose has, of decomposing certain alkali-metal salts, and of appropriating the metallic oxide therefrom, is the basis of various novel mordanting processes, of which the application of a glycerine solution of the sodio-chromic oxide is the most important. The chrome mordant is most valuable for the fixation of cœrulein, alizarin blue, gallein, gallocyanin; also of the newly-discovered Victoria blue (presumably an a-naphthyl-tetramethylrosaniline). Researches in connection with the application of this colouring matter have led to the discovery of the elegant method of chrome mordanting, which consists in padding with a mixture of an alkaline bichromate, sodium thiosulphate, and magnesium acetate. At ordinary temperatures these salts do not react, but on steaming decomposition sets in, and the chromic oxide is fixed in the fibre.

Manganese mordants are employed for the production of various fast shades, by a subsequent dyeing process, which consists in boiling the mordanted fabric in a solution of a salt of an aromatic amine. As a result of the oxidising action of the oxide and its combination with the product of oxidation, a series of extremely fast shades may be obtained. In this way a black may be obtained from aniline sulphate; a red brown from naphthylamine sulphate. The result is susceptible of numerous modifications in its mode of application, some of which are mentioned by the author. He then proceeds to review the growth in the practice, which has now become general, of "preparing" cloth to be printed, by a treatment with Turkey-red oil. The efficacy of this mordant or prepare is entirely referable to the ricinoleic acid, which is its chief constituent. In connection with the subject of mordants, the author alludes to the experience of the dyer that one colouring matter may act as a mordant for another—*e.g.*, Millar's canarin for almost all the aniline colours; alizarin red for saffranin, alizarin violet for methyl violet. This has an important bearing on the practice of "topping" colours.

In his review of progress in the department of colouring matters, indigo is treated first. Mention is made of C. Kœchlin's method of discharge by means of a neutral chromate, printed topically, and afterwards decomposed in a bath of sulphuric acid, containing in addition oxalic, the latter serving to arrest the action of the chromic acid. By incorporating with the chromate a suitable mixture of albumen and a pigment, the latter is fixed by coagulation of the albumen at the same time that the indigo is discharged. By employing aluminium chlorate as a discharge, the fabric may be mordanted at the same time for alizarin, etc. Another process with the same object consists in padding with red prussiate and printing with sodium aluminate. The topical synthesis of indigo from nitrophenylpropiolic acid, the outcome of Bæyer's well-known researches, is a process that remains for the present very restricted in its application. The alkali-glucose reduction method of Schlieper and Baum is favourably noticed. The observation that the alkaline mixture used in this process destroys the adhesion of an alizarin lake to the cotton, is the basis of an indigo blue print upon a Turkey-red ground. It is only necessary to pass through sulphuric acid of 7° B., to complete the topical removal of the alizarin. Mention is also made of the method of reserving alizarin red by means of tartaric acid; also of reserving aniline black by means of the sulphocyanides. In speaking of the colouring matters which have been proposed as substitutes for indigo, the author mentions (1) indophenol. This body, which resembles indigo in giving a colourless reduction product, labours under the disadvantage of being sensitive towards acids, by which it is coloured red, and, in fact, decomposed; hence it has received only a limited application. In the printing of woollen goods, however, it is of more value in consequence of its peculiar property of acting, in the reduced state, as a discharge, upon the azo ponceaux. This is the basis of a method of producing very pretty effects in indophenol blue upon red grounds, and one which the author believes to be destined to a wide application. (2) The colouring matter, as yet uninvestigated, produced by the action of potassium bichromate upon logwood extract, in presence of the bisulphites. It gives, with acetate of chrome mordants, blue shades resembling indigo, but greyer in tone. They are, however, not very fast. In violet colouring matters the only recent introduction of importance is gallocyanine, a body of both basic and acid properties, produced by the action of nitrosodimethylaniline upon gallic acid. It can only be fixed by means of chrome mordants. It is extremely fast. In combination with berry liquor it gives indigo shades.

Cœrulein and gallein, colouring matters discovered by Bæyer, which remained for long unnoticed, are now being extensively used. The former is the basis of the new olive greens, which are extremely fast, and easy of production. The mordant is a mixture of acetate of chrome and sodium bisulphite. The indulins are now commonly used for the production of grey shades; they require a tannin mordant.

In red colouring matters, alizarin continues to be almost exclusively used. Great hopes are, however, entertained of the use of the new product azarin, the bisulphite compound of an azo colouring matter resulting from the action of β-naphthol upon didiazodioxysulphobenzide. A new feature, in this compound, is the presence of the SO_3H group, and the consequent absence of the tendency to sublimation. It is easily fixed by means of alumina mordants, and gives fast shades.

The author then mentions a group of colouring matters which appear to unite directly with cellulose, and are, therefore, fixed without the intervention of a mordant; these are flavophenin, canarin, a product of oxidation of hydrosulphocyanic acid, and the so-called Congo red.

Lastly, of new yellows, the author mentions auramine, an ammonia derivative of tetramethyldiamidobenzophenone; and flavaniline, a base belonging to the quinoline series; and also a pigment yellow produced by steaming a mixture of cadmium nitrate and sodium thio-sulphate.

—C. F. C.

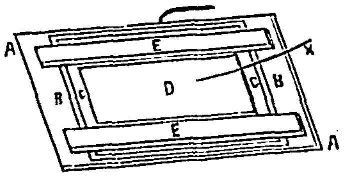
Process for Bleaching Woven Fabrics. J. B. Thompson and J. P. Rückman (D. R. P., Cl. 8, No. 26,839, March 6, 1883). Dingl. Polyt. J. **252**, 392.

Linen yarns and fabrics are first boiled for three hours in a dilute solution of potassium cyanide, washed, and again boiled in the cyanide. Cotton does not need this treatment unless it is more or less greasy, and even then the solution of cyanide must only be half as strong as for the linen, and only two hours' boiling is necessary. For the remainder of the bleaching process, the reader is referred to this Journal, vol. ii. 411, and vol. iii. 291.

—E. G. C.

On the Discharge of Indigo-blue and Turkey-red by Electrolysis. F. Goppelsroeder. Dingl. Polyt. J. 253, 431.

It had been found by Scheurer that indigo-blue was attacked even by moist chlorine gas far too slowly for the discharge process described by Persoz to be a practical success; if, however, the fabric dyed with indigo-blue or Turkey-red were printed with a thickened alkali solution, Scheurer found that the colour of the parts so printed was discharged immediately the fabric was exposed to the action of chlorine gas. In the author's earlier experiments (*Ibid.* 253, pages 245 and 381, 1884) the discharge of these colours was effected by employing as electrolytes solutions of nitrates or chlorides, together or singly, and either neutral or acidified with sulphuric acid. On passing the electric current through a dyed fabric printed or saturated with such solutions, the colour was discharged at the positive electrode, and in the case of neutral solutions being employed caustic potash or soda was set free at the negative electrode. The author was then led by Scheurer's discovery to employ as electrolyte a solution of sodium chloride rendered alkaline with either sodium or potassium hydrate, and found that the evolution of chlorine at the positive electrode was more vigorous than when the electrolyte was neutral or acid. If a fabric dyed indigo-blue or Turkey-red, saturated with a solution of sodium chloride or potassium nitrate, and caustic potash, be laid upon a platinum plate forming the negative electrode, and a platinum wire or plate forming the positive electrode, brought in contact with the upper surface, either colour is immediately discharged. In his experiments the author had recourse to the simple arrangement here figured. Upon the caoutchouc plate A the platinum plate B forming the negative electrode is laid, and upon this, the red or blue fabric C previously saturated with the electrolyte. Between B and C from eight to sixteen folds of similarly saturated white cloth may be interposed if desired. Upon the dyed fabric C the platinum plate D forming the positive electrode is placed, and glass plates E serve to protect the portion of the cloth not covered by D from the action of the gases evolved at the positive pole. This process may also be found of value for the development of various colours.—W. D. B.



H. Köchlin's Chrome Mordant with Colouring Matters. Dingl. Polyt. J. **255**, 447.

The cloth is twice taken through a mordant consisting of one volume of chrome acetate 16°, three parts water, two parts NaOH solution 38° and $\frac{1}{8}$ part glycerine. The cloth is left several hours after mordanting and then thoroughly washed. The dyeing was made with the addition of different substances by way of comparative tests, such as alum, alum and chrome alum, and chrome alum alone, tin crystals, and glue. One part of the sample was steamed and the half of each soaped at 60°. The following results were obtained :—

1. The best effect produced by the addition of an equal weight of chrome alum. Bismarck brown, Coupier's grey, Victoria blue (this requires twenty times the weight of chrome alum), cochineal, eosin B., alkali blue.

2. The best effect produced by the addition of 25 per cent. of tin crystals. Ponceau 3 R., Bordeaux S., rose Bengal, rocceline, eosin, orange II., indigo-carmine, cochineal and Persian berries.

The colours in all cases are brightened by steaming. Those which under these conditions resist soaping are Bismarck brown, malachite green, Victoria blue, gallocyanine, cochineal, logwood, alizarin, nitro-alizarin, Persian berries, quercitron, cœruleine, eosin and rose Bengal. The following three colours give a negative result: Auramine, saffranine, alizarin blue. Ponceau 3 R. dyed with an equal weight of chrome alum on wool after steaming resists soaping at 75°.—J. B. C.

A New Red Discharge on Vat Blue. A. Scheurer. Dingl. Polyt. J. **255**, 452.

A mixture of aluminium chloride and bichromate of potash thickened with starch is printed on the indigo-dyed cloth and then steamed for a minute in a Mather & Platt machine. It is washed, dyed in alizarin, and soaped. The red is somewhat dull, due probably to the products of decomposition of indigotine, which become fixed with the alizarin lake. This method resembles that of G. Saget, who proposed a mixture of aluminium chloride and finely-powdered manganese dioxide.

—J. B. C.

Printing Blue Grounds upon Woollen Fabrics. O. Breuer. Dingl. Polyt. J. **255**, 534.

To obtain, by printing, a uniform blue ground upon woollen fabrics, the author advises the addition to the colour of fifteen per cent. of ammonia. When Blue 5 B. is employed, the best results are obtained by decomposing this colouring matter in aqueous solution, with an acid, and redissolving the blue precipitate obtained, with ammonia. The printing colour is composed of two kilos. of precipitated blue dissolved in 350 grammes of ammonia, together with 500 grammes of glycerol, and 1100 grammes of dry gum senegal. The employment of ammonia is chiefly to be recommended when working with aniline colours; it exerts, however, a beneficial influence in conjunction with nearly all the artificial dyestuffs, even those—the ponceaus, for instance—upon which it has little specific action.—W. D. B.

The Fixing of Various Artificial Dyestuffs by Means of Ferro- or Ferricyanides. Dingl. Polyt. J. **256**, 42.

Certain artificial dyestuffs being precipitated by prussiates, C. Reber (*Bulletin de Rouen*, 1884, 768) makes use of this property for fixing colours on vegetable fibres. For instance, if a solution of aniline violet be added to a solution of a yellow or red prussiate, a complete precipitation of the dyestuff takes place, probably as the ferro- or ferricyanide of methylrosaniline. The same is the case with fuchsin, methylene blue, etc. To carry out this process, the tissue is mordanted with a solution of prussiate. After drying, the dyestuff is printed on it, allowed to hang for 24 hours, steamed, washed, and soaped. Methylene blue and violet produce the darkest and fastest shades with red prussiate, whereas fuchsine yields the most beautiful colours with the yellow salt.

In order to employ the mordanting power of ferro- or ferricyanides for dyeing tissues previously printed upon, the author precipitated zinc ferro- and ferricyanide on the fibre, which compound, indeed, attracts the dyestuff forming permanent lakes. Thus prussiates may in certain cases take the place of tannin, a very desirable matter from an economical point of view.—S. H.

Manufacture of Antimonial Compounds suitable as Mordants in Dyeing and Printing. Wetter. Eng. Pat. 5887, April 3, 1884.

If alkalis, carbonates of the alkalis, or alkaline earths are allowed to act on sugar, gum, dextrin, or other carbohydrates, the products thus obtained give, with antimony, compounds which are soluble in water without decomposition. For instance, a solution of grape sugar is treated with a strongly alkaline substance until no further diminution of the free base takes place. The mass is then acidulated with a strong acid, and digested with antimonious oxide or any antimonial compound adapted to introduce into the liquid simultaneously with the necessary antimony a certain quantity of strong acid; such compounds are the trichloride, the sulphate, etc. In any case, however, that degree of acidity should be imparted to the liquid which causes the absorption of a maximum of antimony. The salts formed from the acid introduced into the liquid, and from the base contained in the same, are removed by filtration or crystallisation. The antimonial liquids resulting from these operations may be directly used in dyeing and printing textile materials, or they may be concentrated by cautious evaporation, or converted into a dry state.
—S. H.

Process of Bleaching Webs or Yarns of Vegetable Textile Materials. William Mather, Manchester. Eng. Pat. 471, January 13, 1885.

This invention consists in applying the operations referred to in the inventor's specification (2641, May 28, 1883), Thompson's specification (595, February 3, 1883), and Cross's specification (4984, Nov. 30, 1880), in combination, and as successive steps of one process.

What is claimed is "in bleaching, by a continuous process, webs or yarns of vegetable textile material, the combination of the operations of saturating with alkaline or equivalent solutions (*i.e.*, sulphite of soda, etc.), steaming, saturating with bleaching liquor, and exposing to the action of carbonic acid gas."—H. A. R.

VII.—ACIDS, ALKALIS, AND SALTS.

The Decomposition of Ammonium Sulphate by Sodium Sulphate under the Influence of Heat. G. Blattner. Dingl. Polyt. J. **255**, 253.

Carey and Hurter have patented a process for the preparation of ammonia from ammonium sulphate, which consists in the decomposition of that salt by means of sodium sulphate at a high temperature, the products of the reaction being free ammonia and acid sulphate of soda, which latter, mixed with common salt and heated, will give hydrochloric acid and neutral sodium sulphate. The author now records experiments he has made with the object of determining the degree of accuracy with which the reaction expressed thus:

$$(NH_4)_2SO_4 + Na_2SO_4 = 2NaHSO_4 + 2NH_3.$$

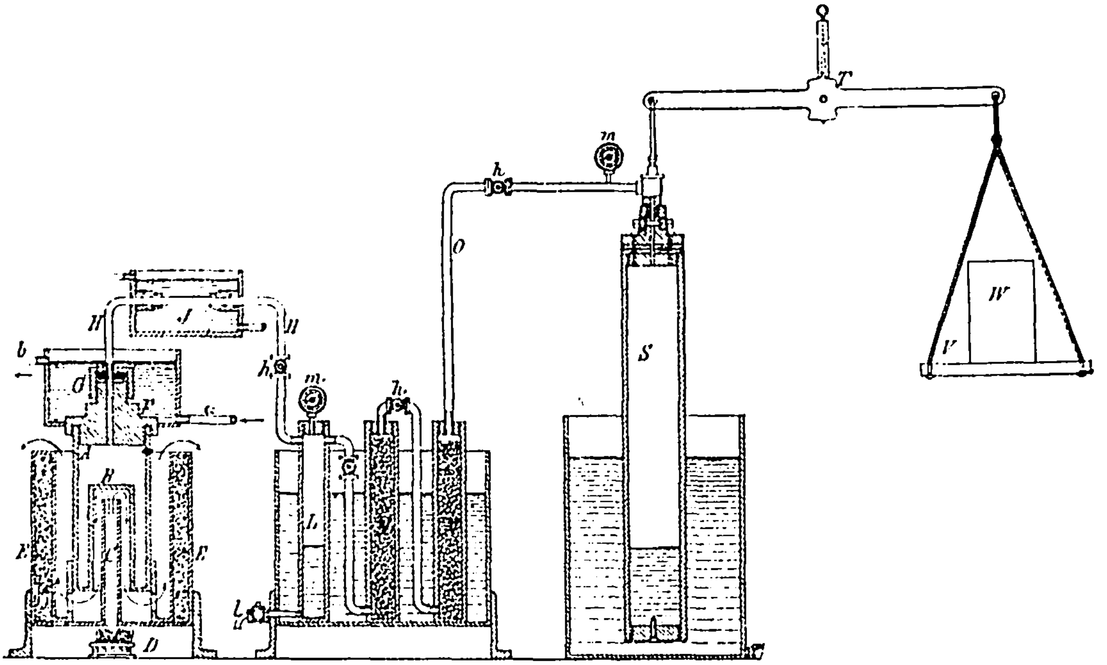
takes place. In the experiments recorded the substances in different proportions were heated to various temperatures in a retort connected with two conical flasks, containing dilute sulphuric acid, for the absorption of ammonia evolved, a stream of air being passed through at the end of each operation. The ammonia which remained in the body of the retort, and also as a sublimate of bisulphite upon the neck, was driven off by soda ley, and estimated. The amount of ammonia lost, probably due to decomposition and liberation of nitrogen, was determined by subtracting the total amount found from that employed in each experiment.

No.	Experimental Details.	Ammonia obtained. Parts per 100 of that taken.		
		Evolved as NH_3.	Remaining in the body of Retort and sublimed.	Lost.
1	10*g*. $(NH_4)_2SO_4$ in solution + 15*g*. Na_2SO_4	58·5	27·1	14·4
2	10*g*. ,, ,, 13*g*. ,,	69·4	14·2	16·4
3	10*g*. ,, ,, 13*g*. ,,	67·8	17·0	15·2
4	40*g*. ,, ,, 60*g*. ,,	65·7	20·6	13·7
5	20*g*. ,, ,, 50*g*. ,,	50·0	37·0	13·0
6	20*g*. ,, ,, 25*g*. ,, gradually heated till fused...	56·3	25·5	18·2
7	20*g*. $(NH_4)_2SO_4$ ground up with 25*g*. Na_2SO_4, rapidly heated	66·6	16·4	17·0
8	Same as 7, but temperature not allowed to rise above 400°	15·4	60·6	24·0
9	20*g*. $(NH_4)_2SO_4$, 35*g*. Na_2SO_4, moistened with water and rapidly heated	58·5	23·0	18·5
10	20*g*. $(NH_4)_2SO_4$ in solution + 23*g*. Na_2SO_4, practically equivalent quantities	68·2	15·1	16·7
11	20*g*. $(NH_4)_2SO_4$, heated by itself	23·1	65·7	11·2
12	20*g*. $(NH_4)_2SO_4$ in solution + 25*g*. Na_2SO_4, temperature not allowed to exceed 400°	28·0	57·8	14·2

These figures show that the best results are obtained by employing approximately equivalent amounts of the substances, but that even then only between 65 and 70 per cent. of the ammonia present is obtained, the remaining 30 to 35 per cent. being partly retained, partly sublimed as bisulphite, and partly lost by decomposition. It may be found possible to prevent or at least lessen the loss of ammonia, by conducting the heating in presence of a current of water vapour.—W. D. B.

Apparatus for the Manufacture of Liquid Carbonic Acid. Dingl. Polyt. J. **256**, 36.

H. & J. F. Beins, Groningen (Germ. Pat. 30,192, 1884), have patented the manufacture of liquid carbonic acid by heating sodium bicarbonate in an apparatus in which the carbonic acid evolved is liquefied by its own pressure. The cylindrical retort A (Fig. 1) is covered with a strong lid F, containing the outlet pipe for the carbonic acid. Its bottom is formed by a pipe B, closed at the top, and open at the lower end, thus exposing a large surface of the retort to the heat of the fire-gases, by compelling them to travel in the direction of the arrows in the diagram. A cooling arrangement protects the packing of the lid from getting destroyed. The carbonic acid given off travels through a cooling vessel J, where the steam is condensed and collects in the cylinder L. Thence it proceeds through two coke-towers, which serve to retain any organic matter, and lastly into the cylinder S, surrounded by cold water or ice. This cylinder is fastened to the beam of a scale for the purpose of filling it with any definite weight desired. The coke-towers stand in a vessel which can be filled with warm water in order to prevent the carbonic acid from being condensed in this part of the apparatus.—S. H.



On the Law of Solution. H. le Chatelier. Compt. Rend. **100**, 50 and 441.

In the first of these communications, the author deduces from the principles of thermodynamics a relation between the solubility of salts, their heat of solution and the temperature. From this equation it is shown that the solubility of a salt which dissolves with absorption of heat, will increase with the temperature and inversely; whilst the solubility of a salt which dissolves with evolution of heat, will diminish with rise of temperature.

—A. G. G.

On the Solution of Magnesium Carbonate by Carbonic Acid. R. Engler. Compt. Rend. **100**, 444.

By estimating the solubility of magnesium carbonate in water at 12° C., charged with carbonic acid at various pressures, the author has obtained numbers from which he calculates the general equation $X^{\cdot 333} = ky$ (where y= quantity dissolved, X= pressure of CO_2 and k= a constant = ·03814);

$$\text{Hence } X = k^3y^3 \text{ or } y = \frac{1}{k}\sqrt[3]{X},$$

that is to say, the quantity dissolved at any fixed temperature is proportional to the cube root of the carbonic acid pressure.—A. G. G.

A Crystallised Hydrate of Phosphoric Acid. A. Joly. Compt. Rend. **100**, 447.

By cooling the mother-liquor from the trihydrate, prismatic plates of a tetrahydrate ($P_2O_5+4H_2O$) crystallise out. This melts at 27° C., *its heat of liquefaction* being 3·64cals. It dissolves in less than 100pts. of water with absorption of heat, in more water with evolution of heat.—A. G. G.

Nitre-earth from Turkestan. N. Ljubawin. J. Russ. Chem. Ges. 617, 1884.

An area of about four square kilometres, south-west of Fort Nukus, on the left bank of the Amu-Darjos, in the Khanate of Khiva, is covered with a layer of nitrate earth of the following composition:—Constituents soluble in water=27·89; Ditto soluble in HCl=17·14; CO_2=5·73; insoluble=48·42. Total 99·18. The part soluble in water contains—

KNO_3	5·52	10·61 total nitrate.
$NaNO_3$	4·05	
$Mg(NO_3)_2$	1·04	
NaCl	12·90	
$CaSO_4$	3·25	
$MgSO_4$	0·66	

—J. T.

Improved Method of Producing Oxide of Strontium or of Barium. E. F. Trachsel, Middlesex. Eng. Pat. 6022, April 5, 1884.

Sulphate of strontium or barium is reduced to sulphide by heating with carbonaceous substances. The sulphide is then heated with oxide of iron or iron in a muffle in presence of air, and hydrate of strontium or barium is obtained from the product by extraction with water.

—G. H. B.

Improvements in the Method of Generating Carbonic Acid Gas, and in Apparatus therefor. J. Magnall and R. S. Lloyd, Middlesex. Eng. Pat. 6179, April 9, 1884.

CARBONIC acid is obtained by the action of dilute sulphuric acid on whiting, the reaction being maintained by agitation.—G. H. B.

Improvements in the Manufacture of Alumina. G. Rosenthal, London. Eng. Pat. 6290, April 12, 1884.

SULPHATE of alumina is heated with its equivalent of magnesium chloride, the reaction being assisted in the later stage by the application of superheated steam. Hydrochloric acid is evolved, and the residue, consisting of alumina, oxide of iron and magnesium sulphate, is lixiviated with water, and the oxide of iron is removed "by magnets."—G. H. B.

Improvements in the Manufacture of Carbonate of Soda. C. Wigg. Eng. Pat. 7525, May 10, 1884.

THIS invention is a combination of the Leblanc and the ammonia soda process, in order to obtain hydrochloric acid as well as carbonate of soda. The process can be resolved into the following stages: 1. Common salt is converted into sodium sulphate, the hydrochloric acid given off being condensed in the usual manner. 2. Sodium sulphate is mixed with a strong solution of ammonia or ammonium carbonate, and the temperature raised to that point at which the maximum of sodium sulphate enters into solution. The operation is performed under pressure in a closed vessel, provided with hollow rotating beaters. 3. The solution is allowed to cool while impurities settle out which can be removed. 4. Carbonic acid is passed into the solution under a pressure of from 15 to 40lb. until all carbonate of ammonia is converted into bicarbonate. Sodium bicarbonate is thereby precipitated and ammonium sulphate enters into *solution.* 5. Sodium bicarbonate is separated from ammonium sulphate by filtration, first through cloth and afterwards through gypsum, so as to ensure that the ammonium carbonate which has not entered into reaction *with sodium sulphate shall be decomposed into ammonium* sulphate. 6. The filtrate is evaporated to obtain ammonium sulphate for sale, or the solution is treated with brine and boiled down. By this means anhydrous *sodium sulphate, free from iron, is precipitated and a* solution of ammonium chloride is obtained, from which the ammonia can be liberated for re-use. 7. The sodium bicarbonate is roasted to convert it into carbonate of soda, carbonic acid being recovered at the same time.

—S. H.

Improvements in Apparatus for Disintegrating and Removing Impurities from Lump Salt. T. Higgin, Liverpool. Eng. Pat. 8266, May 27, 1884.

THE object of this invention is to remove without pulverising the "pan scale" and "sand scale," which the lumps contain, and which are harder than the salt. These can then be removed by sieving. The lump salt is disintegrated by hackling or tearing, as distinguished from crushing or pulverising. The specification contains a large number of drawings, giving the details of the machinery employed.—S. H.

Manufacture of Sulphate of Ammonia and Bye-products resulting from the Process. Kelly and Weigel. Eng. Pat. 8080, June 6, 1884.

KIESERITE *solution is mixed with common salt and the* mixture subjected to a low temperature, when a double decomposition takes place producing sodium sulphate, which crystallises out, and magnesium chloride, which *remains in solution. Ammonia is then passed along* with carbonic acid through the solution of sulphate of soda, thus producing sodium bicarbonate and ammonium sulphate. During this reaction the vessels are kept cool. The bicarbonate is allowed to settle and removed in the solid condition, whereas the solution of ammonium sulphate, which also contains small quantities of sodium bicarbonate, sodium sulphate, ammonium hydrate and carbonate, is subjected to distillation, in order to drive off the volatile ammonium compounds. At the same time, the small quantity of sodium bicarbonate is converted *into* sulphate of soda, which can be removed by crystallisation. Ammonium sulphate is then obtained from the residual solution by evaporation.—S. H.

Improvements in Purifying Sulphuric and Sulphurous Acids and their Salts. J. Meikle. Eng. Pat. 11,866, September 2, 1884.

THIS invention consists in passing sulphuretted hydrogen through the acid or a solution of their salts. The effect is to form a precipitate of sulphides of arsenic or other metals, and this precipitate, which is separated by filtration, may be utilised by burning so as to yield sulphurous acid. Sulphuric acid derived from pyrites and purified by this process is well suited for making sulphate of ammonia of good quality.—S. H.

Improvements in the Manufacture of Chlorine. Ernest Solvay, Brussels. Eng. Pat. 7258, January 15, 1885.

THESE improvements relate to the process referred to in Patent No. 77, January 8, 1877; No. 838, January 25, 1880; and No. 839, January 25, 1880. 1. To prevent the introduction of carbonic acid into the chlorine, the silicious or aluminous substances used are treated with an acid to decompose carbonates, and ignited (with nitrate of soda, if necessary) to remove organic carbon. 2. When the mixture to be decomposed contains chloride of calcium corresponding with the required theoretical quantity of silica and alumina, the matter agglomerates and the apparatus works with difficulty. The addition of more silica or alumina gives the required infusibility, but the material is no longer suitable for the manufacture of cement. This difficulty is obviated by the introduction of a sufficient quantity of the residuum of a previous operation. 3. The air used is purified from carbonic acid and moisture by passing through a series of suitable absorbing vessels.

—A. R. D.

Improvements in the Manufacture of Chlorine and Chloride of Lime. Ernest Solvay, Brussels. Eng. Pat. 7259, January 16, 1885.

THESE improvements relate to the same process as the preceding. To enable the apparatus to resist the action of chlorine the iron or cast-iron is covered with preservative coatings of certain mixtures of soda, clay, and vitrifiable products. To this enamel is sometimes added a fireproof coating which adheres to the metal through the intermediary of the vitrified enamel. The absorption by lime is effected by placing the lime in a layer of suitable thickness on beds of porous mineral matters, such as pebbles, or mineral fabrics, such as asbestos cloth, and causing the diluted chlorine to pass downwards through the same. If the gases pass upwards, channels are soon formed and irregularity ensues.—A. R. D.

Improvements in the Manufacture of Hydrochloric Acid. Ernest Solvay, Brussels. Eng. Pat. 7260, January 16, 1885.

THIS invention relates to the process of decomposing calcium chloride with aqueous vapour in the presence of silica or silicious substances. When this mixture is made in the theoretical proportions the matter agglomerates and the apparatus works with difficulty. The addition of more silica and alumina, while promoting the infusibility of the mixture, renders it unsuitable for the manufacture of cement. The difficulty is obviated, however, by introducing a sufficient quantity of the residuum of a previous operation. Silicious clays, infusorial silica, fossil flour, etc., as well as precipitated silica, are much to be preferred to sand in the above operation.—A. R. D.

Improvements in obtaining Solid Caustic Soda from Fused Caustic Soda. W. Weldon. From A. R. Pechiney et Cie, Salindres. Eng. Pat. 2478, February 24, 1885.

THIS invention consists in a method of obtaining caustic soda in films or sheets, capable of being readily converted into powdered caustic soda. Fused caustic soda is brought into contact with the exterior surface of a revolving hollow cylinder, the heat being carried off by water passed through the interior of the cylinder. In Figs. 1 and 2, A is a hollow cylinder turning on hollow

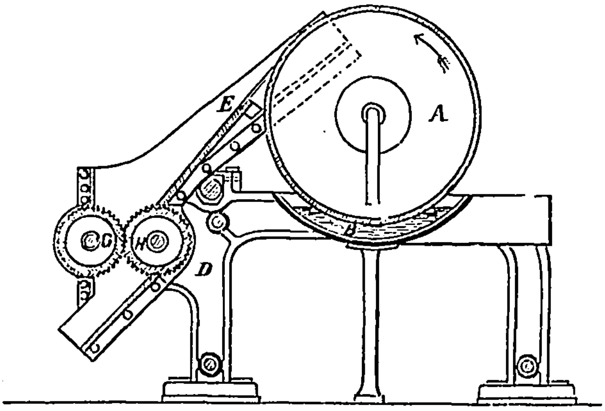


FIG. 1.

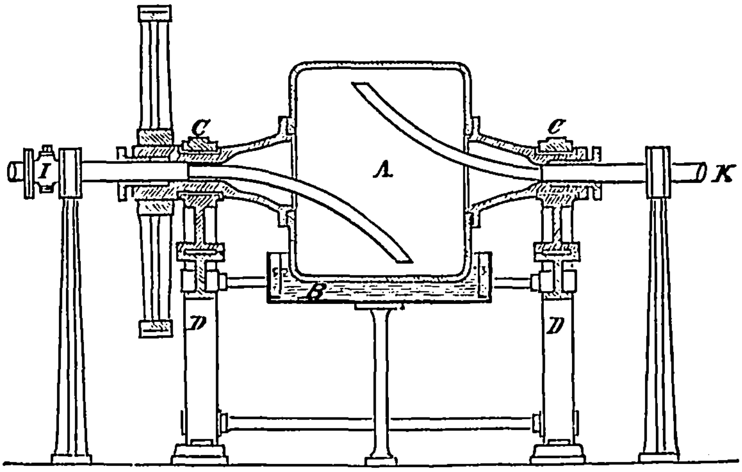


FIG. 2.

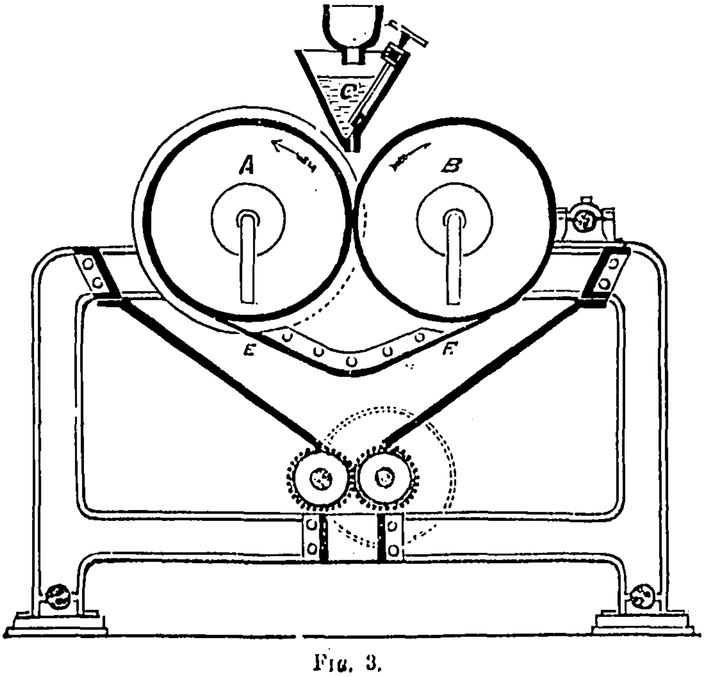


FIG. 3.

axles CC. Water is admitted into A, passing out near the top by the pipe K. Below the cylinder is a small trough B, into which fused caustic soda is fed. As the cylinder A revolves, it dips a little below the surface of the caustic soda and emerges coated with a film of solid caustic soda, which on reaching the scraper E is detached from the cylinder and falls into a conduit, from which it falls between the toothed rollers G and H, which crush it into small fragments. Figs. 3 and 4 represent another disposition of apparatus for the same purpose. There are two hollow cylinders A and B turning in opposite directions. At each extremity of the cylinder A is a flange which overlaps the corresponding extremity of the cylinder B. C is a hopper into which the fused caustic soda is charged. It is provided with a slit at the bottom and a contrivance to regulate the feed at will. The two cylinders are kept cooled by water continually passing through them. E and F are scrapers which detach the solid caustic soda and deliver it between the two toothed rollers.—S. H.

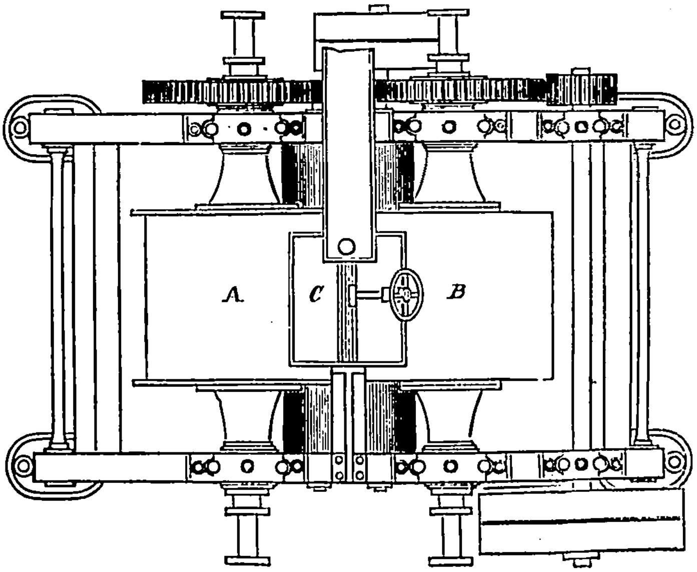


FIG. 4.

The Protection of Nitrate of Ammonia from Deliquescence. W. R. Lake, London. Eng. Pat. 1868, Feb. 10, 1885.

NITRATE of ammonia, used in the manufacture of explosive compounds, is protected from deliquescence by mixing it in a granulated condition with bye-products from the petroleum distillation. It then resembles moist brown sugar, and is not liable to become lumpy or solid.—S. H.

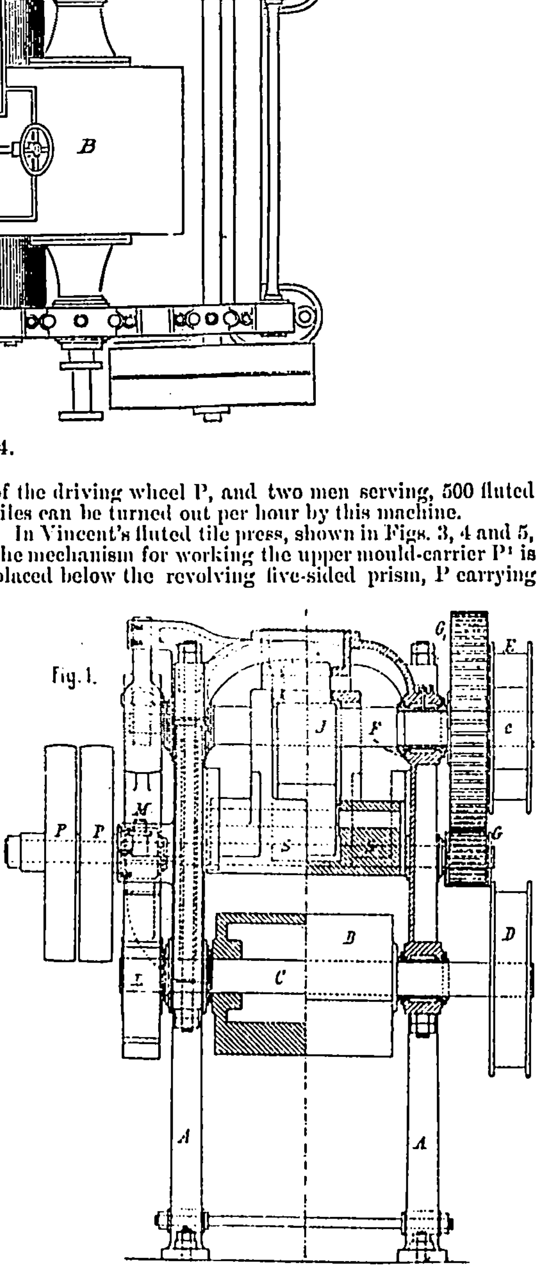
VIII.—GLASS, POTTERY, AND EARTHENWARE.

Recent Improvements in the Manufacture of Earthenware. Dingl. Polyt. J. **255**, 505.

IMPROVEMENTS in Schmerber's press for the manufacture of fluted tiles have been made by Boulet, Lacroix & Company. In Figs. 1 and 2 are shown the arrangements now adopted. The prism B, upon the five sides of which the lower moulds are fixed, revolves upon the axis C, motion being transmitted from P by S, G, G_1, E and D, the two latter being pulley wheels in connection. The revolution of B is regulated by the ratchet wheel L, and catch M. When M is lifted up by means of the cam N upon the spindle F, the prism is free to revolve, but the catch soon falls again, and when the next notch of L is brought beneath M, the prism is brought to rest. By this means the moulds upon B and S are brought to exact and certain correspondence; this was not the case in the original machine. The slide S, which carries the upper mould, is actuated by the cam J. The moulds are of plaster of Paris, and are fixed in iron boxes in the usual manner. With 1 horse-power, 55 or 60 revolutions of the driving wheel P, and two men serving, 500 fluted tiles can be turned out per hour by this machine.

In Vincent's fluted tile press, shown in Figs. 3, 4 and 5, the mechanism for working the upper mould-carrier P^1 is placed below the revolving five-sided prism, P carrying the lower moulds. By the introduction of steam into the interior of M_1 and P^1 the moulds are heated that the tiles may have less tendency to adhere. The flat plates of clay to be fluted are carried on to the lower mould by the endless band G; when the pressing is accomplished a frame A is laid upon the tile and there held by catches C, until by the revolution of P^1 the tile attains a horizon-

Fig. 1.



tal position; on releasing the catches the tile falls off. This arrangement for retaining the tile upon the mould forms the subject of the patent (Ger. Pat. 29,514, May 29, 1884). Magowan and Bassett (Ger. Pat. 28,527, December 19, 1883) adopt the plan of warming the moulds for dishes and such ware, in order to facilitate the removal of the moulded clay, interposing thin sheets of metal or caoutchouc between the mould and the clay. Thus in moulding plates, the lower mould is covered with a thin sheet of metal, by means of which the moulded clay is easily removed from the mould; between the clay and the upper mould is a thin sheet of caoutchouc which can be readily detached from the clay plate after pressing. The Brothers Netzch (Ger. Pat. 28,651, February 5, 1884) have devised the following process for placing clay sheets with rapidity and accuracy upon the mould or wheel for further manipulation. The sheet of clay is first prepared upon a wheel covered with leather, the edge of which is turned down, as shown in Fig. 6, and held firmly between two rings *b*. The inner ring firmly embraces the circumference of the wheel. The clay which projects beyond the edge of *a* is cut off with wire, and the disc lifted from off the wheel by its rings.

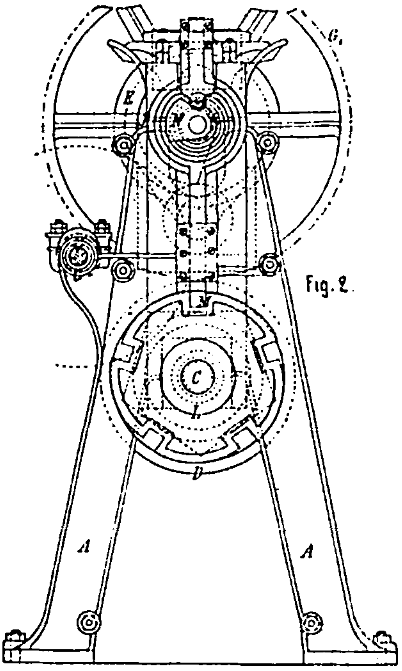


Fig. 2.

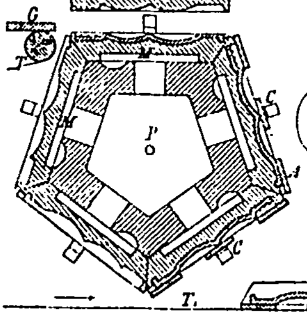


Fig 3

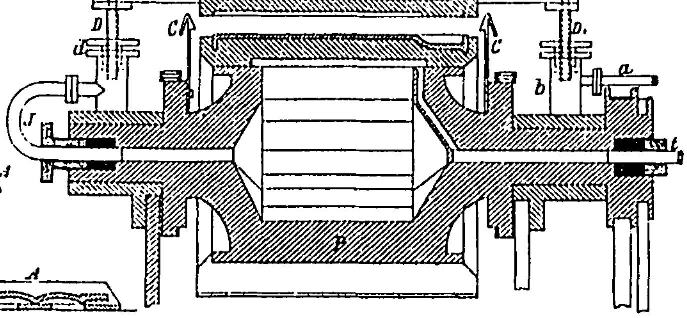


Fig. 4

A second workman, who has to carry the work a further stage, has, near his wheel, the apparatus shown in Fig. 7. A rod S, movable vertically in two guides shown, carries at its lower end an arm E with disc G attached. The counterpoise M is adjusted to maintain S in its highest position until it is required to depress G, which is effected by the cord N and a foot lever. The ring of the leather disc described above is now passed over C, which is fixed in such position in respect to E that by turning the latter round upon S until E is in contact with H, the centres of G and the wheel are in vertical line. G is now brought down to the wheel, upon which,

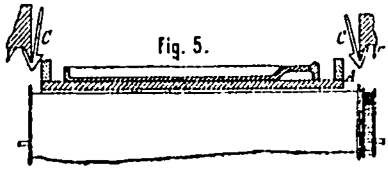


Fig. 5.

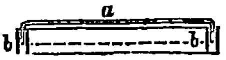


FIG. 6.

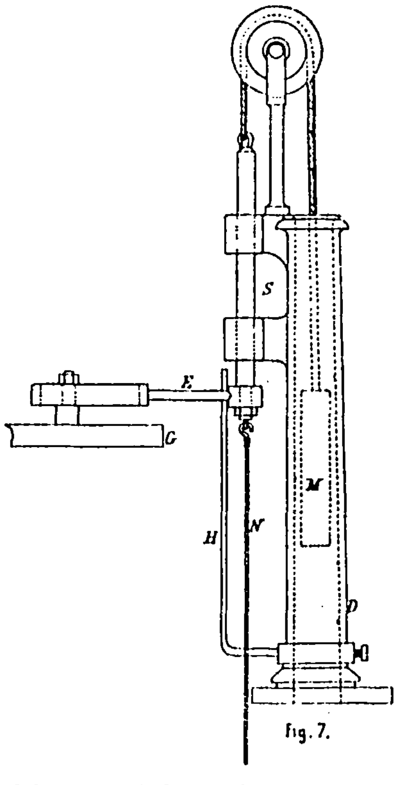


Fig. 7.

on rising, it leaves the clay sheet and disc. The wheel being set in motion, the leather is loosened by a spatula, removed, and the clay manipulated as usual. By means of the machine patented by Gregg (Ger. Pat. 29,693, May 30, 1884) it is intended not only to measure off accurately the requisite quantity of clay for each tile, but also to distribute coloured clay or glazing flux over the two opposite surfaces of each tile.—W. D. B.

Solubility of Glass. E. Bohlig. Zeits. Anal. Chem. **23**, 518.

THE author found that a number of new flasks ordered for laboratory use gave up to 100cc. of boiling water sufficient alkali, in the form of silicate of soda, to neutralise $\frac{1}{10}$cc. of oxalic acid every two seconds. The solubility in this particular case appears to be quite exceptional.—J. B. C.

Improvements in the Manufacture of White Opaque, Opalescend or Semi-transparent Glass and Enamel. John Henry Johnson. From Adolf Zeodesco, Mügeln, Saxony. Eng. Pat. 6899, April 28, 1884.

THE inventor substitutes for phosphate of lime, cryolite-felspar or fluor-spar, or the double fluoride of aluminium and alkali in conjunction with the usual ingredients for producing opacity in glass—an alkaline fluoride either with or without the conjoint use of a substance containing alumina. He regards as an important point the advantage of this mixture as a substance of easy production, either by adding to fluoric acid the requisite quantity of an alkali or its carbonate, taking care to have an excess of alkali. Or by treating the aluminate of an alkali with fluoric acid till all the alkali is fixed so that hydrate of alumina is precipitated, and after calcination a mixture of alkaline fluoride and alumina will result. Although mentioning the use of alumina and alkaline fluoride for the production of opalescence in glass or enamel, the inventor claims only the use of the fluoride of an alkali in conjunction with materials employed for producing such result.—T.

IX.—BUILDING MATERIALS, CLAYS, MORTARS, CEMENTS.

Adulteration of Portland Cement. R. and W. Fresenius. Zeits. Anal. Chem. **24**, 66-71.

THE name Portland cement should be confined to the product obtained by heating together lime and clay in definite proportions and then finely powered. When this product is mixed with more than 2 per cent. of foreign matters, such as gypsum, the authors contend that the name "Portland cement," if applied to the mixture, is fraudulent. Whether a cheap substance, without any influence on the durability, etc., of the cement is used as an adulterant or a substance added to improve the quality, the name "Portland cement" cannot rightly be employed for the mixture. The authors consider that the nomenclature needs revision and seek the co-operation of cement manufacturers for this purpose. Their tests furnish a ready means of distinguishing the pure article and substitutes or mixtures (*Zeit. Anal. Chem.* 23, 175).—S. R.

An Improved Process and Apparatus for Treating Wood, chiefly Designed for the Preservation of Railway Sleepers. W. R. Lake, London. From E. L. Collings and C. F. Pike, U.S.A. Eng. Pat. 833, Jan. 20, 1885.

THIS invention consists in the treatment of wood by first steaming it, then drying it by radiated heat and subjecting it to the action of an antiseptic material vaporised by radiated heat. The apparatus and the mode of procedure are described in detail in the specification.
—D. B.

X.—METALLURGY, MINING, Etc.

Process for the Extraction of Cobalt and Manganese. Herrenschmidt and Constable. Dingl. Polyt. J **252**, 392.

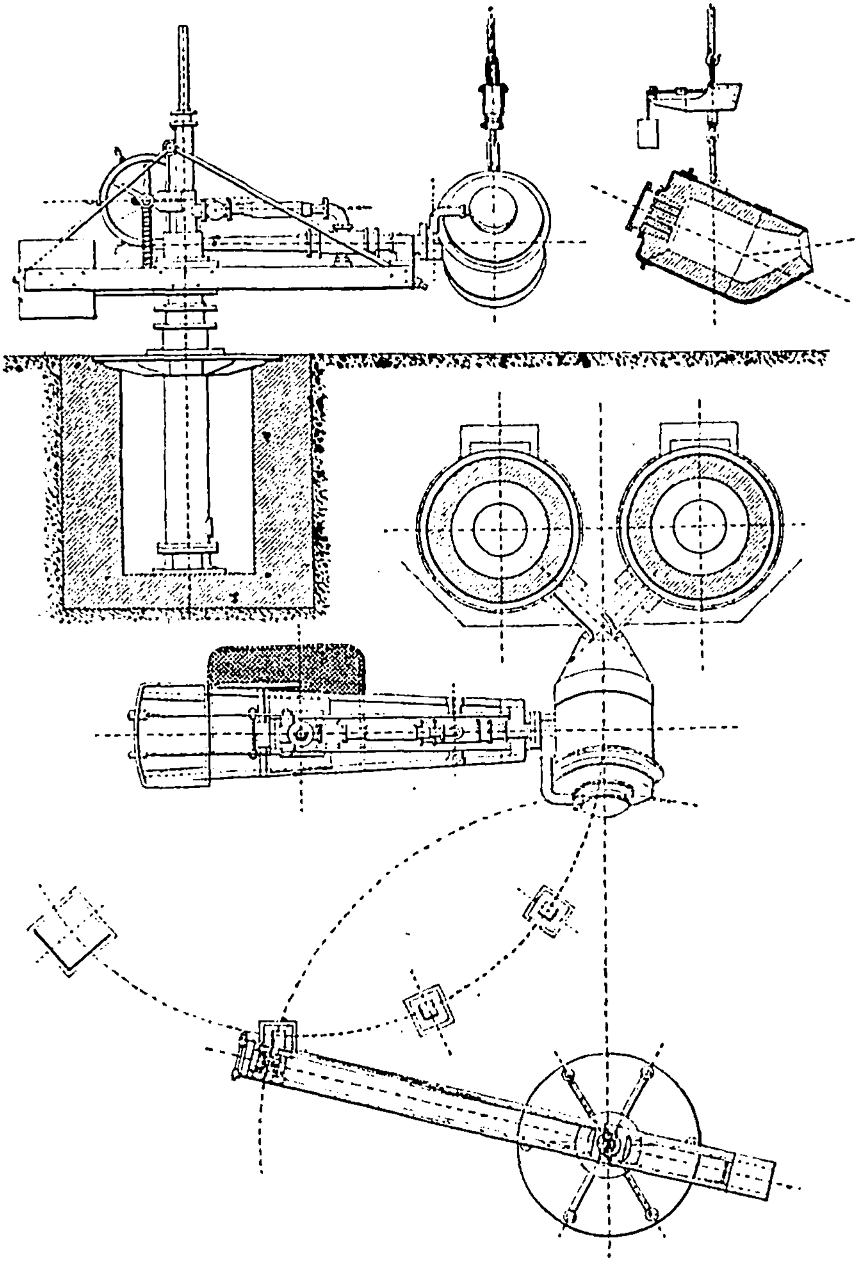
ORES containing cobalt or manganese are pulverised and boiled for half-an-hour with water and sufficient ferrous sulphate to convert the oxides present into cobaltous and manganous sulphates, which go into solution; this is decanted from the residue, the proto-salts being afterwards oxidised into per-salts.—E. G. C.

On the Preparation of Aluminium. Dingl. Polyt. J. **253**, 426.

H. D. GADSDEN, London (Ger. Pat. 27,572, August 8, 1883), mixes corundum, bauxite, and the like with about 10 per cent. of sodium or potassium fluoride and the same amount of calcium fluoride. The whole is calcined, ground to a powder, mixed with 10 per cent. of wood-charcoal or carbonaceous matter, balled and transferred to a retort or muffle, in which it is burnt. The spongy mass obtained is removed to a second retort, and exposed to the action of a stream of chlorine gas; the aluminium chloride which is formed, is distilled into a receiver, having been, if necessary, previously freed from iron by leading the vapour through a tube filled with iron turnings. To effect the reduction to aluminium of the chloride so obtained, the latter is transferred to a fire-clay retort, connected by a leading tube with a second similar retort, in which a mixture of 20 parts of sodium carbonate and 16 parts of charcoal, cr an equivalent amount of other carbonaceous matter, and 5 parts of lime or chalk, is heated to such a temperature that sodium vapour distils over into the aluminium chloride, reducing the latter to the metallic state. The aluminium collects at the bottom of the retort, and may be drawn off and cast into bars.—W. D. B.

Progress in Metallurgy. Dingl. Polyt. J. **255**, 375.

Siemens-Martin Process.—J. Riley read an important paper on the arrangement of plant, at the Chester meeting of the Iron and Steel Institute (see *Journ. Iron and Steel Inst.* ii. 1884, pp. 443-452). With a modern 12-ton Siemens furnace, "good work" requires that 140-150 tons of ingots shall be made weekly per furnace. In 1880 experiments were made with a basic lining, but great difficulty was caused by fluxing at the lines of division of the basic and acid material, and the experiments were stopped. At Blochairn a row of twelve 15-ton furnaces was erected, and the arrangement finally adopted for casting was as follows:—The furnaces were marked A to M, the last one being capable of holding 25 tons, the others being 15-ton furnaces. On a railway in front of, and parallel to, the furnaces runs a small locomotive which moves a special carriage conveying a ladle for the steel, and a mould for the slag. A 20-ton hydraulic lift is erected between furnaces E and F, and over the railway. In front of this is a 20-ton casting crane which does not lift, but turns on its axis, and has a ram for turning over the casting ladle after each cast. This crane is placed in the centre of a semi-circular casting pit, 40ft. in diameter and 3 feet deep. Around the casting pit are three ingot cranes, and between the cranes are two sets of soaking pits, each with six cells of differing capacity for ingots of 30 or 60cwt. each. The two outer cranes set the moulds in the pit, strip the ingots and deposit them in the soaking pits. The centre crane lifts the ingots from the soaking pit and places them vertically on the tipping carriage at the end of the feed rollers of the cogging-mill. A hydraulic ram tips the ingot easily and without shock on to the feed-rollers of the mill. When a furnace is ready for tapping, the contents are run into the ladle on the carriage, the whole of the steel and slag being run into it, while the latter overflows into the slag mould. Being brought under the lift the ladle is raised and tipped automatically, so that the steel flows into the casting ladle. The charge is now transferred to the moulds in the usual manner. The time occupied in tapping the furnace, transferring and casting is fourteen to fifteen minutes. After standing twenty minutes the ingots are stripped and placed in the soaking pits, where they remain about fifty-five minutes; altogether the slab is finished in about ninety-five minutes, five minutes going to cogging and shearing.



Basic Process in Siemens-Martin Furnace.—T. Gillot read a paper before the American Institute of Civil Engineers. The hearth was formed of lime 58 parts, magnesia 24, silica eight, alumina and iron oxide 10 parts. This material was burnt at a strong white heat, pulverised, and mixed with hot tar. The mass is at once stamped in position with red-hot stamps. Between the basic mass and the acid wall is inserted a layer of magnesia and bauxite. After 72 hours' firing, a layer of freshly burnt dolomite was spread over the hearth. About 10 per cent. of refractory stone is mixed with the dolomite to bind it. The furnace so prepared was charged with freshly burnt lime, then with pig-iron, and lastly with iron and steel scrap. The amount of lime was $\frac{1}{10}$ to $\frac{1}{20}$ of the pig charged. In three to four hours the pig is melted, and a liquid slag can be run off. In three hours more the scrap is melted and more slag is run off. If necessary the slag may be thickened by the addition of lime or iron ore, according as the proportion of pig to scrap is large or small. After the boiling period, a test is taken, and if the result is satisfactory the whole of the slag is run off. Five per cent. of hematite pig is now added, and a violent boiling results for 15 minutes; after 15-30 minutes more the charge is tapped off. The hearth is repaired, and in one to four hours is ready for another charge.

Bessemer Converters.—P. Lamberty (Ger. Pat. 29,571, April 30, 1884) proposes to make the bearing ring hollow, to serve as air chest, and from it the air is passed obliquely downwards through the side of the vessel.

W. Hupfeld, discussing the same subject, remarks that for rolled articles which do not require to be further treated, such as rail, a moderate amount of silicon up to 0·5 per cent. is not injurious in presence of an equal amount of manganese; but for articles richer in carbon, or which have to bear reheating, the silicon should be reduced as much as possible. Although a metal can be produced in large vessels with 0·12-0·20 per cent. carbon, 0·03-0·06 silicon, 0·25-0·4 manganese, 0·035-0·05 phosphorus, and 0·01-0·03 sulphur with the greatest certainty, still a method of further reducing the silicon in Bessemer metal is desirable. According to F. Müller the metallic bath only reduces silicon from a rich silicious slag at a high temperature. Hence, according to Hupfeld's view the lower temperature of a small Bessemer apparatus offers a partial solution of the problem. Successful results have been obtained at Prävali as was previously the case at Avesta. *P. v. Tünner* writes on the subject, but concludes that large blows must prove most economical.—J. T.

Combination of Carbon with Iron. Walther Hempel. Ber. **18**, 998.

It has been shown that iron can take up carbon from its gaseous compounds, such as carbonic oxide, cyanogen, and hydrocarbons, at temperatures only moderately high.

The author describes experiments which prove that a much higher temperature is required for absorption of solid carbon by iron if oxygen is excluded. This temperature varies with the modification of carbon used, and is lowest for diamond. The lowest temperature at which a plate of wrought iron absorbs carbon when covered with powdered diamond and heated in a stream of nitrogen is 1160° C., with pure charcoal this temperature is between 1385° and 1420° C. If iron is fused in the electric arc in an atmosphere of nitrogen it is converted into white iron.—A. G. G.

Improvements in the Manufacture of Metallic Compounds or Alloys. John Radford, Shipley, Yorkshire. Eng. Pat. 3233, February 13, 1884.

The improvement consists in the addition of chromium, either as such or as chrome iron or chrome steel, to bronze, brass, gun metal, white metals, or similar alloys. Nickel and cobalt are sometimes employed in addition to the chromium.—W. G. M.

Improvements in the Collection and Treatment of Flue-dust. H. Barclay and R. Simpson, Cumberland. Eng. Pat. 3498, February 18, 1884.

The deposition of the flue-dust from furnace gases is assisted by means of trays placed in the flues, and also by washing the gases with water. Alkaline salts are obtained from this dust by extraction with water and evaporation of the solution, and the residue may be used as a raw material for iron smelting.—G. H. B.

Improvements in Electro-plating Chromium Alloys. Thomas Slater, Bayswater. Eng. Pat. 5245, March 21, 1884.

The anodes of chromium alloy may be prepared by heating chromium compounds with charcoal in a closed crucible, and pouring upon the reduced mass $2\frac{1}{2}$ parts of fused copper, and, subsequently, 1 to $1\frac{1}{2}$ parts of molten tin; and then granulating, re-fusing, and casting in moulds of the desired shape. These are next used as anodes in a solution, preferably made by dissolving 1lb. of potassium cyanide and a like quantity of ammonium carbonate in a gallon of water, heated to 150° F., until a good deposit of alloy is formed upon the cathode, when the bath is ready for use. To finish the plated articles, they are coloured with a bath of chloride of tin (6 to 8 parts), chloride of copper (20 to 25 parts), bichromate of ammonia (10 to 15 parts), chloride of platinum (6 to 12 parts), cyanide of potassium (25 to 30 parts), and water (101 to 110 parts). A very moderate current only is necessary.—W. G. M.

Improvements in Extracting Gold and other Metals by Amalgamation, and Apparatus therefor. Ewald Fischer, Silesia, and Max William Weber, London. Eng. Pat. 5834, April 2, 1884.

According to this invention, ground or crushed ore mixed with water is stirred with mercury for a sufficient time to effect the amalgamation with it of the precious metals. The ore is then discharged, leaving part of the metal in the mercury, but carrying away with it a considerable amount of the mercury and amalgam in a finely divided condition. These tailings are then subjected to the action of an electric current at the same time that they are stirred or agitated, which effects a complete condensation of the metallic and amalgam particles. Suitable apparatus is described in the specification, and four drawings.—B.

Improvements in the Purification of Iron Ores and of Iron by the Basic Process. Wm. Ph. Thompson, Liverpool. From R. Sohliwa, Dortmund, Germany. Eng. Pat. 7404, May 8, 1884.

The invention refers to a method of eliminating sulphur and silicon from iron ores and iron whilst under treatment by the Thomas and Gilchrist process.

The bottom of a converter lined with magnesia and lime is covered with a mixture consisting of two parts of pulverised anhydrous carbonate of soda, and one part of pulverised carbonate of lime. When the blow has begun a sufficient quantity of the mixture is added to make the total weight of the mixture present to be in the proportion of one part of carbonate of soda and one-half part of carbonate of lime to two parts of the silicon present in the whole charge.

After formation of the silicates, the fluid slag is carefully removed, and for every three parts of sulphur in the crude iron, ten parts of carbonate of soda (without lime) are added to the molten metal, the blowing being continued until desulphurisation has been effected. The slag now formed is again carefully removed, and another ten parts of carbonate of soda (without lime) are added for every three parts of phosphorus contained in the crude metal, and the blow finished. The slag resulting from this final blow is added to that first obtained, and is afterwards treated as phosphatic slag containing phosphoric acid soluble in water.—B.

Improvements in the Manufacture of Spongy Iron, and in Apparatus employed therefor. Gustav Bischof, London. Eng. Pat. 8093, May 22, 1884.

The inventor treats iron ores or products containing iron in an oxidised state with hydrogen and carbonic oxide and anhydride in such a way as to heat and deoxidise them at one operation.

For this purpose steam is decomposed over incandescent coke, the resulting gases consisting essentially of hydrogen, carbonic oxide, and carbonic anhydride, and some free steam. The latter having been removed by cooling and condensation, the remaining gases are strongly heated and passed into the lower part of an inclined retort into the upper end of which the ore is fed, and allowed gradually to descend to the lower part. Here it comes in contact with the mixed gases, and is reduced to the metallic state by the hydrogen contained in them. The spongy iron so obtained is discharged into a receptacle and cooled before exposure to the air.

The remaining gases which pass upwards through the inclined retort, deprived of the greater part of their hydrogen, but still combustible, are met by a current of air, and burned inside the retort. In this way they heat the oxide of iron preliminarily to its contact with hydrogen, and make any external heating in most cases unnecessary. The process is carried on at a low red heat.—B.

Improvements in the treatment of the Oxides of Iron obtained either from Iron Pyrites or from other sources, so as to render them more suitable for employment in the Blast or other Furnace. James Mason. Eng. Pat. 8679, June 6, 1884.

The inventor proposes to remedy the difficulty hitherto found in the use, for blast and other furnaces, of iron oxide in a state of dust or minute particles. He mixes such oxides with a considerable amount of water, and places the wet mass, whether in lumps or moulded, upon a bed of fuel in a kiln, filling in the interstices and covering up with fuel. After the firing has commenced further layers of the mass and fuel are gradually added, and the process continued until the kiln is filled, when the burning is completed, and the mass allowed to cool. The small particles will then be found to have been strongly agglutinated by the heat, and the mass be fit for use as required. The specification is accompanied by a sheet of drawings, showing a kiln arranged for the purposes of the patent.—B.

An Improved Method of Amalgamating or Separating Metals from their Ores. Jonathan Aldous Mays, London. Eng. Pat. 8295, May 27, 1884.

In this process the mixture of ore and water is fed in thin streams beneath the surface of flowing mercury, by means of a series of nozzles, themselves stationary or moving in a direction opposite to that of the mercury. This is effected by confining the mercury in a circular metallic pan, in which it rotates, either through the rotation of the pan itself or by the action of internally revolving arms. The nozzles are fed through an annular chamber, connected with a supply tube of sufficient height to allow of the contained mixture of ore and water overcoming the pressure of the column of mercury in the pan.—W. G. M.

An Improved Process of Preparing or Treating Copper. Alex. Melville Clark, Middlesex. From Wunibald Braun, Frankfort-on-the-Maine, per Wirth & Co. Eng. Pat. 8355, May 28, 1884.

A current of hydrochloric acid gas is passed through the melted copper, and thus all metallic oxides (including that of copper), and all metals, which are capable of decomposing the gas, are removed as volatile chlorides. A very pure metal of low electrical resistance is obtained, and may be cast free from blowholes in moulds filled with any neutral gas, such as hydrochloric or carbonic acid, or lighting gas. The process of poling copper may be partially or entirely superseded by this process.—W. G. M.

Process for the Reduction of Iron Ores and Production of Iron and Steel. Fredk. Siemens, Westminster, and Joseph G. Gordon. Eng. Pat. 8490, May 31, 1884.

Finely pulverised iron ore, intimately mixed with carbonaceous and fluxing materials, is introduced (in the form of bricks or enclosed in iron vessels) into an open hearth regenerative furnace of the Fredk. Siemens type (Eng. Pat. 5677, 1883). There it is reduced, and the metal and slag fused as fast as they are formed, solely by the radiant heat of, and not by direct contact with, the flame. The fluid metal and slag separate by gravity.—W. G. M.

Improvements in Processes for obtaining Copper, Silver, and Gold from Copper Matte or Regulus. Wm. Aristides Vérel and Peter Stewart, Tharsis Sulphur and Copper Co., Glasgow. Eng. Pat. 8562, June 4, 1884.

This patent is intended to render the ordinary wet process available for the treatment of regulus. The matte is crushed fine and mixed with its own weight of sodium chloride, and then, to the extent of 5 or 6 per cent., with the usual mixture of burnt pyrites and salt; the whole mass is then calcined, and subsequently treated as in the usual wet process.—W. G. M.

Improvements in obtaining Gold and Silver from Auriferous and Argentiferous Sulphide of Antimony Ores. Jas. Simpson and Edward Wm. Parnell, Liverpool. Eng. Pat. 13,074, October 2, 1884.

In the treatment of sulphide of antimony ores with solution of the sulphide in alkali or alkaline sulphide as by Patents 11,827 and 11,828, 1884, the whole of the silver, and the greater part of the gold, remain in the residue, a small portion only of the gold passing into the solution as sulphide. To obtain the precious metals from the residue, the sulphur is to be removed by roasting, or by any wet method; and the desulphurised mass treated by amalgamation, chlorination, or any other suitable process. The gold is recovered from the sulphide solution by precipitating a small quantity (2½%) of the antimony sulphide by acid or other means, which quantity will carry with it the whole of the gold; the latter may then be removed by any known process.
—W. G. M.

Depositing Magnesium and its Alloys. F. W. Gerhard, Wolverhampton, and Jas. Smith, Stoke-upon-Trent. Eng. Pat. 16,651, December 19, 1884.

Ammonia sulphate of magnesia, prepared by crystallising together 228 parts magnesium sulphate and 132 parts ammonium sulphate, and dissolving in 35,000 parts water, is best used at from 150° to 212° F. For white metal, a nickel anode; for magnesium bronze, a copper anode is used; in the latter case the bath consists of magnesium ammonium sulphate 360, potassium cyanide 550, ammonium carbonate 550, water 35,000.
—W. G. M.

Improvements in the Treatment of Sulphide of Antimony to obtain Chlorides, Oxychlorides, and Metallic Antimony. John Hargreaves, Widnes, and Thomas Robinson, Widnes. Eng. Pat. 7206, February 4, 1885.

The bulk of the free acid contained in the solution of chloride of antimony, prepared by dissolving ores containing sulphide of antimony in hot hydrochloric acid, is either removed by evaporation or neutralised with lime or magnesia, and the concentrated solution of chloride of antimony is slowly mixed with water in a wooden or stone vessel provided with stirring apparatus. The precipitated oxychloride of antimony is allowed to settle, the supernatant liquor decanted, and then sufficient milk of lime added to convert the oxychloride into oxide, the mixture meanwhile being kept at about boiling heat. The oxide of antimony is thoroughly washed from calcium chloride, dried, mixed with alkali and free carbon, and reduced to metal in any convenient way.
—A. R. D.

XI.—FATS, OILS, AND SOAP MANUFACTURE.

The Extraction and Treatment of Fats. Dingl. Polyt. J. **253**, 415, 1884.

For the extraction of oil from sesame seeds, maize and the like, Bang and Sanguinetti have recourse to mineral oil, boiling between 40 and 70°, purified by repeated agitation, first with sulphuric acid of 66° B., then with two or three per cent. of fuming sulphuric acid. The naphtha thus purified leaves no odoriferous matter, either in the oil or in the seeds. The form of apparatus patented (Austro-Hungarian Patent of July 5, 1883) is shown in Fig. 1. The ground seeds are contained in the metal basket c, which is inserted in the boiler R, the cover n being then placed on c, and the lid c tightly fitted on R. The purified naphtha is run in through t, displaced air escaping at s. After about fifteen minutes' digestion, the second boiler is connected at r, with an exhaust arrangement, and the naphtha from the first boiler drawn over, allowed to remain in contact with the contents of R (II.) for fifteen minutes, drawn over into a third apparatus, and thence so on through several more vessels R, until by the process of repeated diffusion a mixture of oil and naphtha is obtained, containing very little of the latter.

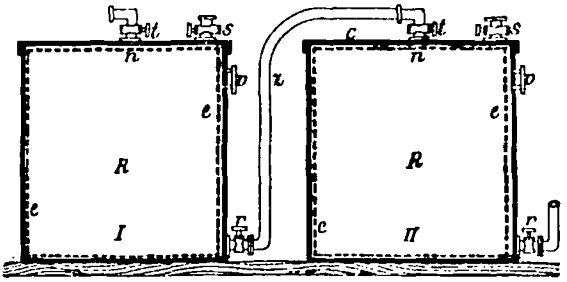


Fig. 1.

Successive quantities of fresh naphtha are passed through the apparatus, and when the contents of the first of the series of vessels R are found to be free from fat, connection is made at r with a condenser, and superheated naphtha vapour passed in at s, until by the temperature of the mass in R, it is evident that all the naphtha retained by the seeds has been vaporised. The stream of superheated vapour is now shut off, and the basket c taken from R, the contents being in a completely dry state. If it be desired to remove every trace of naphtha from the seeds, this can be effected by the application of dry steam.

According to O. Korschelt (Ger. Pat. 27,321, September 4, 1883), the saponification of fats by superheated steam is effected advantageously only between the narrow limits of 310° and 315°, the glycerine being decomposed at higher, and the reaction being incomplete at lower, temperatures. If the fatty matter be finely subdivided, however, the limits of temperature may be somewhat wider. For the purpose of subjecting finely divided fatty matters to the action of superheated steam, and at an efficiently regulated temperature, the apparatus shown in Figs. 2 and 3 has been patented. The fat is heated in A to about 100°, and passes thence into the winding tube a lying in the bath B, filled with an alloy of 100 lead with 6 or 4 tin, kept as nearly as possible at its melting point 290°. The oil attains to a temperature of about 300°, this being observed by interposing between B and the tower a vessel h, through which the oil flows, fitted with tubes open above, but closed below, containing lead melting at 334°, and the above lead-tin alloy melting at 289°. The pipe a enters the tower C near the bottom, and passes up to within a small distance from the top, where it is bent over and fitted with a circular spreader, of diameter slightly less than that of the tower. The oil rises to this height either by reason of the elevated position of A, or by the aid of a force pump interposed between A and B. The tower C is constructed of sheet iron, or of cast-iron plates, surrounded with masonry, and is filled with earthenware balls. Superheated steam is passed in at the bottom by c, its temperature being observed by aid of g, an arrangement similar to h, described above. The products of the decomposition of the glycerides are carried away by the current of steam through d at the top of the tower, and are condensed. The temperature within the tower may vary from 250° to 400°; it is not advisable, however, to allow it to sink below 280°. Any undecomposed fat is run off from the bottom of the tower by the pipe i.—W. D. B.

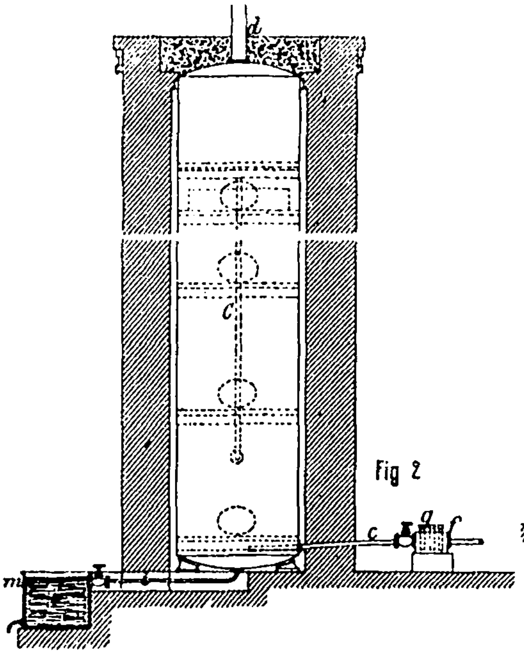


Fig 2

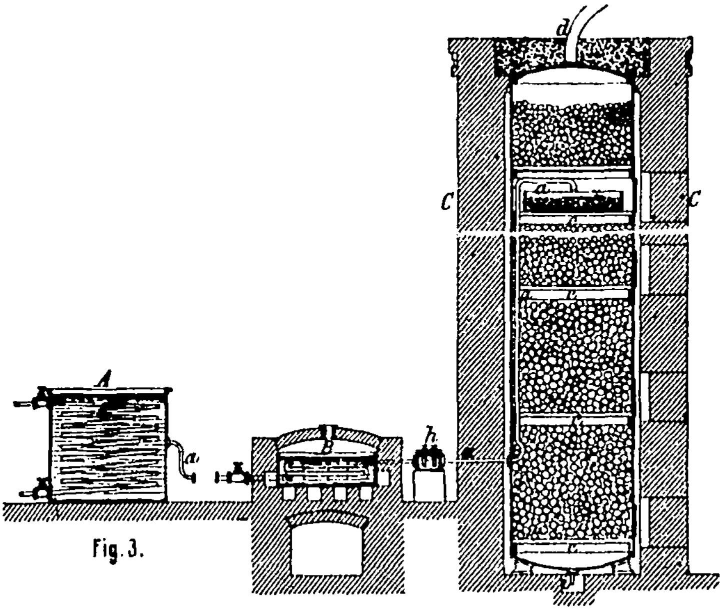


Fig. 3.

The Production of Grape-stone Oil. J. v. Jobst. Dingl. Polyt. J. 255, 450.

As a fatty oil is said to be extracted from grape stones in Italy, the author attempted, although unsuccessfully, to express the oil. By extracting with carbon bisulphide or petroleum ether, about 10 per cent. of a thick green oil was obtained, which is nearly decolourised by filtering through charcoal. It possesses an unpleasant smell and taste, and solidifies at 11°. On standing in the air it becomes rancid and thick. These properties exclude its use for culinary purposes, and also as a lubricating oil. It may be employed for lighting; but better for the manufacture of soap. The production of the oil in small quantities is unprofitable, and is confined to certain districts, where it is chiefly employed for lighting purposes.—J. B. C.

Preparation of Curd Soap. F. Eichbaum. Dingl. Polyt. J. **255**, 539.

A GOOD curd soap with silvery fracture may be prepared by boiling 700kg. tallow, with soda lye of 15°, to a clear jelly, introducing 450kg. of palm-nut, and 100kg. of cocoa-nut oil, with the requisite amount of caustic lye of 25°, and boiling the whole until a clear jelly, free from froth, is obtained. After about two hours any scum upon the surface is removed, and the soap salted out or precipitated respectively with salt solution of 20°, or caustic soda lye of 40°.—W. D. B.

XII.—PAINTS, VARNISHES, AND RESINS.

On the Manufacture of White Lead. Dingl. Polyt. J. **252**, 372.

ACCORDING to an American Patent (292,119, January 15, 1884), M. Roth and G. Sylvester pass an electric current through a solution of sodium acetate, using leaden plates as the poles. Acetate of lead is formed at the positive pole, and caustic soda at the negative pole. By the mutual reaction of these compounds, lead hydrate is produced, which is afterwards converted into white lead by the action of carbonic acid gas. E. V. Gardner (D. R. P., Cl. 22, No. 25,239, August 19, 1882) heats lead with graphite, carbon, platinum, or some other substance electro-negative to lead, in a solution containing 1 part acetate of lead, or 1 part nitric acid, in 40 parts of water, or acetic acid in 24 parts of water. Basic lead salts, or lead oxide, or lead suboxide, will in this way be formed. After one or two hours, the lead is taken out, and the fluid rendered capable of being used again by the addition of acetic or nitric acid. If the operation be performed in chambers, the lead is so placed as to be in contact with the carbon or platinum. Ozonised air should be conducted into the chamber, or generated therein by electric discharges. The apparatus employed is shown in the figure.—E. G. C.

Analysis of Vulcanised Caoutchouc, including the estimation of the Sulphur. B. Unger. Zeits. Anal. Chem. **24**, 167-174.

THE method adopted for estimating the sulphur in caoutchouc by heating the sample with copper oxide and sodium carbonate, and then, after dissolving in aqua regia, precipitating the sulphate formed with barium chloride, is untrustworthy. Antimony sulphide and sulphur only give a sulphate, when soda is present as well. The presence of alkalies does not retard the reaction, but prevents the dissociation of the sulphuric acid at the high temperature necessary for the complete combustion. The antimony sulphide used in the English manufacture contains free sulphur (6·30 per cent.) and gypsum (9·62 per cent.). The sulphur of the gypsum present is not available for vulcanisation, so that the determination of the amount of sulphur in the caoutchouc should be accompanied with the percentage of calcium as well as that of the antimony present. The author recommends the following processes:—

1. *Sulphur.*—About ·5grm. of the sample is cut into fragments and heated with 12grm. copper oxide and 2grm. soda, in which the amount of sulphate present has been previously determined. The copper oxide is prepared by heating well-washed copper carbonate. The caoutchouc is added to the crucible in small quantities at a time—the progress of the reaction and the amount of heat can be regulated by observing the smell emitted which should be pleasant and aromatic (not unlike kümmel). The product is dissolved in aqua regia and evaporated over a water bath to dryness to render the antimonic acid insoluble, and to obtain a solution which can be easily filtered. After treating with water, filtering and washing, the filtrate is diluted to 600cc. The sulphate is then precipitated with barium chloride in the usual way.

2. *Antimony.*—About 1·5grm. of the substance is cut up into fragments and heated with 10grm. of crystallised sodium sulphide. The product is triturated with water and filtered, and then the filtrate is heated with hydrochloric acid. The mixture of antimonious sulphide and sulphur is weighed on a tared filter paper, and the result checked by heating a known portion with excess of sulphur and reweighing.

3. *Calcium.*—About 2·5grm. are incinerated in a porcelain dish, and the temperature gradually raised to redness. The ash is heated with strong hydrochloric acid and allowed to stand for 24 hours. The solution is diluted with water to precipitate the antimony as oxide, and ammonia is added in excess to complete the precipitation. To the filtrate ammonium oxalate is added, and the calcium is then weighed as carbonate in the ordinary manner. The results are calculated out to give the percentage of—Sb_2S_5, $CaSO_4.2H_2O$, and free S in the caoutchouc. The amount of rubber present is given by difference.

Another mixture used in England to give a black vulcanised indiarubber contains—

66·55	Talc (with FeO ; Al_2O_3 ; H_2O).
21·10	Litharge.
4·30	Zinc Oxide.
8·05	Sulphur.
100·00	

With lead present as in this last case, when the sample is heated with copper oxide and soda, the sulphate formed is chiefly lead sulphate, which can be dissolved out from the residue with ammonium acetate. Part of the lead sulphate and the zinc are estimated in the copper solution, and a second sample is heated first in the air and then with soda to find the percentage of talc present.—S. R.

Improvements in, and in Apparatus for, the Manufacture of Lead Salts for making White Lead. W. V. Wilson, Middlesex. Eng. Pat. 6464, April 17, 1884.

THE apparatus described has for its object the formation of solutions of basic lead salts by causing finely divided metallic lead to be exposed alternately to the action of air and steam, and then of a solution of neutral acetate or nitrate of lead. It consists of a revolving drum provided internally with shelves, on which the lead is raised and allowed to fall on a perforated cylinder also provided with shelves, and revolving in the centre of the drum. Air and steam or hot air is sent through the drum at the central bearings, which are made hollow, and the drum is kept half-full of solution of acetate or nitrate of lead. The treatment of the basic lead solution takes place in another vessel.—G. H. B.

Improved Means and Apparatus for Facilitating the Dissolution of Metallic Lead, or of Oxide of Lead in the Manufacture of White Lead. A. W. Anderson, London. Eng. Pat. 5166, December 18, 1884.

A HOLLOW wooden drum rotating slowly on a hollow axis has a number of radial shelves projecting more or less towards the centre, and on the axis of the said drum are secured other radial shelves projecting towards the circumference. The hollow axis is perforated and provided with a partition so that a hot blast of air may be admitted to the drum on the one side of this partition, and escape through the perforations on the other side. Finely-divided lead is placed in the drum, which is then partially filled with suitable solvent liquid, and caused to rotate. By this means the lead, being alternately exposed to the action of the solvent liquid and the blast of hot air, is rapidly dissolved. When the liquor is sufficiently concentrated it may be drawn off for the precipitation of the lead with carbonic acid gas, or this precipitation may be effected in the drum itself.—A. R. D.

XIV.—AGRICULTURE, MANURES, Etc.

Improved Process and Apparatus for the Utilisation of Human Excreta, Drainage from Stables, and the like. W. P. Thompson. From J. R. Bryden, Georgetown, British Guiana. Eng. Pat. 6441, April 17, 1884.

THIS invention is based on the fact that if sewage matter be received in reservoirs, in which it can be kept out of contact with outside air and in contact with soil, a natural chemical process ensues resulting in the transformation of the excrements into a liquid manure containing nearly all the valuable nitrogenous matter of the sewage. By natural filtration through the soil, animal and vegetable remains become disintegrated, the decomposing matter being finally converted into nitrates. A series of underground closed reservoirs with earth bottoms receive the sewage which is afterwards utilised for manurial purposes.—E. G. C.

Improvements in the Manufacture of Phosphates. F. W. Harbord, Wolverhampton. Eng. Pat. 7562, May 10, 1884.

THE object of this invention is to obtain phosphates practically free from iron from the slags resulting from the Thomas-Gilchrist process, or from other phosphatic materials containing iron. The phosphate is finely ground and freed as far as possible from metallic iron. It is then treated with very dilute acid, care being taken to dissolve as much phosphoric acid as possible. The solution is separated from the residue and steam, and air blown through it, in order to peroxidise the iron. A solution of a ferrocyanide is then added in just sufficient quantity to precipitate all iron in form of Prussian blue, which is separated and treated to recover the ferrocyanide. The phosphatic solution may be worked up for free phosphoric acid or precipitated with chalk or lime, if precipitated phosphate be required.—S. H.

Improvements in Treating Phosphates of Calcium or Lime. P. M. Justice. Eng. Pat. 7963, May 20, 1884.

THIS invention relates to the treatment of phosphates of lime which may be in a dry or humid state, and consists in first calcining and decarbonating them, and subsequently subjecting them to the action of cold or hot water or steam, and as soon as they are cold subjecting them to a high temperature. By this means the material is more thoroughly decarbonated, and a more perfect division or disintegration obtained, the material being left in a condition to permit of the easy elimination of the lime by any of the ordinary means.—S. H.

XV.—SUGAR, GUMS, STARCHES, Etc.

Extraction of Sugar from Molasses. Dingl. Polyt. J. **253**, 421.

IN the extraction of sugar from molasses by his strontia process, Scheibler now avoids the formation of bistrontium saccharate by adding to the mother liquors and washings separated by filtration from the monostrontium saccharate prepared as originally described, further amounts of molasses and strontium hydrate in the proportion requisite for the production of monostrontium saccharate. The mother and wash liquors are thus worked up repeatedly until they contain so much non-saccharine matter as to prevent further advantageous treatment. After 6 or 8 successive operations, the sugar remaining in the liquors need not amount to more than 4 or 3 per cent. of that originally present in the molasses treated. The ammonium chloride osmose process as carried on at the sugar refinery at Haussy is described by Dureaus in *Journ. des Fab. de Sucre*, 1883, No. 46. The syrup separated from product I is mixed with 1 per cent. of ammonium chloride, heated to 100° and subjected to osmose with 10-12 parts of water at 75°. After this treatment the syrup has a density of 11° B. (hot), the proportion of sugar to mineral matter being 12 or 14 to 1, whilst the original proportion was 5 or 6 to 1. The osmosed syrup is added to fresh juice. In the *Zeit. deut. Ver. Rübenzucker-Ind.* 1884, p. 85, details are given of the working of Stutzer's precipitation process, depending upon the precipitation of calcium saccharate from an alcoholic solution of the molasses. It has been shown by experiment, that the precipitation of the calcium saccharate takes place most readily if the lime has previously been slaked in spirit, whilst the precipitation is effected both slowly and incompletely if the lime be introduced as powder. From this it appears that the lime when hydrated combines more readily with sugar than when in the form of oxide, especially in a saccharine solution containing alkali. The hydroxides of barium and strontium behave similarly.—W. D. B.

Sugar-Beet Culture. Dingl. Polyt. J. **255**, 354.

FOR the valuation of sugar-beet seed, Sempotowski (*Deutsche Zucker-Industrie*, 1884, pp. 272 and 1280) allows 5 grammes to germinate at 20° in a wooden box filled with sifted garden soil. From 1 gramme of very good seed 81 to 112 sprouts may be expected, of good from 55 to 80 sprouts; a sample of seed giving less than 40 sprouts per gramme is to be regarded as bad. The best results in cultivation trials, made in Saxony in the year 1883 by Maercker (*Neue Zeit. für Rübenzucker*, 1884, 12, pp. 142 and 257), were obtained with Dippe's improved "Little Wanzlebener Beet." The best nitrogenous manure for the potato and sugar-beet is, according to Wagner, not ammonium sulphate but Chili saltpetre. That the former has under certain conditions—such as moist, warm weather, or on soils rich in lime and humus and especially rapid nitrification—given as good results as the latter is not denied. On the other hand there is no undoubted case on record where manuring with Chili saltpetre was followed by a worse crop than that with ammonium sulphate. P. Dehérain (*Compt. Rend.* 1884, 99, p. 920) found that the "Vilmorin" variety, unmanured, yielded a juice of which 100 parts contained 19 parts of sugar, and that, with favourable weather, a plot manured yielded beets equally rich in sugar. The advantage gained by the application of manure was the increase in total crop: 1 hectare, unmanured, gave 29,700kilos.; manured with stable dung, 35,000kilos.; with stable dung and Chili saltpetre, 43,000kilos. of beet. From experiments conducted at the experimental station at Gembloux as to the best method of applying artificial manures, it appears that upon sandy-clay soils a surface dressing after harrowing is not sufficient, since the absorptive powers of such soils are so great as to prevent, even in wet years, the plant food from finding its way to the tap-root. The manures are most advantageously dug in for some depth in the spring-time. The results of Corenwinder's experiments as to whether the sugar beet derives the carbon of its sugar contents mainly from the atmosphere, are instructive. The plants were cultivated partly in pure sand containing the necessary salts, partly in a manured soil, partly in the open. The following results were obtained:

	Total Weight of		Composition of Root.		
	Leaves.	Root.	Water.	Sugar.	Ash.
Sand	270g.	490g.	80·8	12·26	0·98
Manured Soil	2560g.	1145g.	83·8	10·60	1·16
Open Field ...	......	......	83.2	9·00	0·91

The beet grown in pure sand thus produced 61 grammes of sugar, for the formation of which 31cc. of carbonic acid must have been assimilated on an average per diem.
—W. D. B.

Analysis of Honey. E. Sieben. Dingl. Polyt. J. **255**, 441.

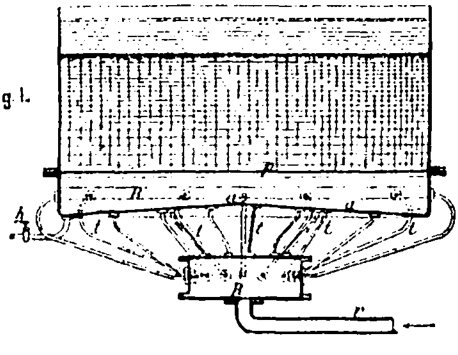
THE amount of cane-sugar in the samples was determined by Meissl's method by difference before and after inverting with dilute hydrochloric acid. The method of Sachsse for estimating dextrin compounds present by boiling with hydrochloric acid, and thus converting them into grape-sugar is unreliable. A method for estimating the grape-sugar in honey is to boil with an excess of sixfold normal hydrochloric acid, which destroys the

levulose. After neutralising with caustic soda the grape-sugar is determined according to Allihn. The difference in the amount of grape-sugar given by this method and that of Sachsse-Fehling varied between −2·44 and +2·29. The analyses show that in the majority of cases grape-sugar and levulose are present in nearly equal quantities. In most samples cane sugar is absent, the highest percentage found being about eight. The amount of water varied between 16 and 25 per cent., and the amount of non-sugars, including wax, between 1 and 9 per cent. The author gives a useful review of the methods for detecting starch-sugar syrup used in the adulteration of honey. The sulphuric acid reaction is condemned, as pure honey also contains larger or smaller quantities of this acid. Precipitation with alcohol cannot be employed as not only is the dextrin of the starch-sugar thrown down, but also albumen and other substances present in the unadulterated article. The method proposed, which will detect the smallest trace of this adulteration, is based upon the following facts. Solutions of honey, in which the cane-sugar present has been inverted, when heated with *a slight* excess of Fehling's solution contains no longer any substance which on further heating with hydrochloric acid forms sugar. Starch-sugar syrup, on the other hand, by a similar treatment yields for every *100grm.* syrup *nearly 40grm. grape-sugar.* 14grm. honey are dissolved in 450cc. water and heated with 20cc. of half normal hydrochloric acid for half an hour on the water bath, by which the cane sugar is inverted. It is neutralised, made up to 500cc. so that a 2 per cent. invert sugar solution is obtained. 100cc. Fehling's solution is titrated with this solution. It is filtered through asbestos, washed with hot water, neutralised with concentrated HCl, and ·1 volume of concentrated HCl added, and then heated for one hour on the water bath. The solution is nearly neutralised with concentrated NaOH solution and made up to 200cc., 150cc. of the filtered solution is heated with 120cc. Fehling's solution and 20cc. water, and the grape-sugar estimated according to Allihn from the weighed copper.
—J. B. C.

Apparatus for the Manufacture of Starch. Dingl. Polyt. J. **256**, 35.

FOR the manufacture of rice-starch H. Mack, Ulm (Ger. Pat. 30,256, 1884), proposes treating rice with caustic soda, and blowing air under pressure at the same time through the liquid in such quantities that the latter assumes the appearance of a boiling liquid. For this purpose air enters through many small tubes *t* (Fig. 1) into a tank B provided with a false bottom *p*, on which finely ground rice is spread, whereas the tank is filled with a very dilute solution of caustic soda. After finishing the operation the caustic liquid is run off at *h*, and the rice-starch formed allowed to drain on the perforated bottom. The inventor states that by the aid of large volumes of air the rice softens quicker, and yields from 6 to 8 per cent. more of the finished product, thus saving time, plant, and chemicals.

Fig. 1.



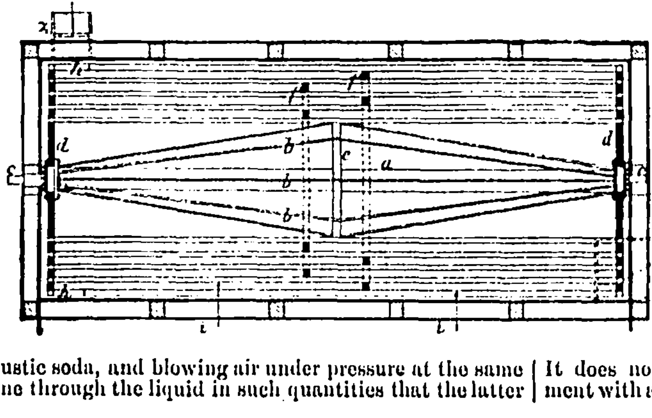


Fig. 2.

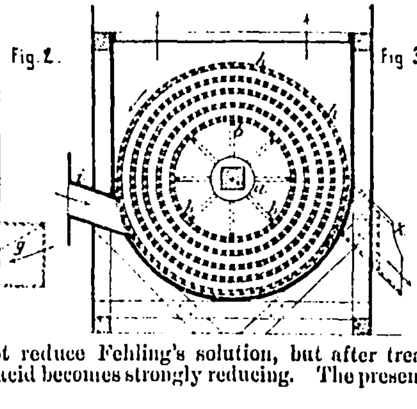


Fig 3.

L. Fehrmann, Parchim (Ger. Pat. 29,600, 1884), uses for drying starch a drum revolving round a shaft *a* (Figs. 2 and 3). Discs *d* are screwed to the journals c, and serve as a support for the ends of a great number of iron rods. From the centre of each disc also issue the distributing rods *b*, which meet in the middle, where they are fastened to a wheel *c*. Starch enters the apparatus at *g*, and being seized by the outside row of rods it is carried to the highest point of the drum, falls to the next series of rods and so forth, until it arrives at the bottom only to make the same way over again. The drum inclines towards the outlet, and the revolution thus causes the mass to proceed gradually to the outlet *z*. When it arrives here it is dry, and in a finely divided state. The warm air necessary for drying travels in a channel along the whole length of the drum, and escapes at the top, deprived of its heat, into an air-filter, which retains the powder mechanically carried away.—S. H.

The So-called Plus-Sugar. E. von Lippmann. Zeits. dent. Zucker Ind. 1885.

THE author has found needle-shaped crystals of a sugar, so-called plus-sugar, in molasses, which appear to be identical with the substance examined by Tollens (Ber. 18 [1], 26). Being readily soluble in dilute alcohol and water the crystals were separated from the molasses by means of absolute alcohol. Analysis gave numbers corresponding to the formula $C_{12}H_{22}O_{11}+3H_2O$, which agrees with Tollen's formula. A 10 per cent. solution shows a specific rotation $[\alpha]_D=105$ at 19°. The sugar is insoluble in absolute alcohol and ether, is not precipitated by lead acetate except in an ammoniacal solution, and is not acted on by solutions of alkalies or alkaline earths. It does not reduce Fehling's solution, but after treatment with acid becomes strongly reducing. The presence of this sugar is the cause of the occurrence of needle-shaped crystals in raw sugar extracted from molasses, and of the high rotation of such sugars.—D. B.

Improvements in the Extraction of Sugar from Molasses and other Saccharine Products, and in the Quality of Alkaline Salts resulting therefrom. Victor Daix and Antoine Louis Possoz, Paris. Eng. Pat. 7235, May 5, 1884.

This process consists in osmosing molasses or other saccharine products, and then precipitating the sugar from the exosmose waters, molasses, and other residues, as sucrates of lime, strontia, or baryta. The mother waters which have been exhausted of sugar are concentrated, and nitrate of soda is then added in sufficient proportion to transform the chlorides and other potassic salts into nitrate of potash, which can be more easily obtained from the liquors than the original potash salts.
—A. W.

A New or Improved Process for obtaining Sugar from Saccharine Substances. C. D. Abel. From Dr. Ludwig Harperath, Cologne. Eng. Pat. 7982, May 20, 1884.

This process consists in treating molasses, diffusion juices, or other saccharine solutions with burnt natural dolomite or a calcined mixture of magnesia and lime, so as to produce a magnesian lime saccharate, which is more permanent than the lime saccharate, and is not so subject to retrogressive action. As little or no development of heat takes place on the admixture of the dolomite with the saccharine solution, no special cooling appliances are required. The magnesian lime saccharate, in contradistinction to the poly-basic lime saccharates, always forms without cooling, and even at temperatures above 50° C., but not between 30° and 50° C. Cooling is only required when the atmospheric temperature reaches or exceeds 30° C., as the reaction will not commence above that temperature. The purification and decomposition of the saccharate is effected by known methods.
—A. W.

Improved Apparatus for Osmose Treatment of Saccharine Liquors. J. Imray, London. From H. Leplay, Paris. Eng. Pat. 8502, May 31, 1884.

This apparatus is a modification of the well-known "osmogene" of Dubrunfaut. In conducting the process of osmosis in Dubrunfaut's apparatus, the molasses or other liquor under treatment becomes gradually more dilute as the process goes on, and this increased dilution is accompanied by a corresponding decrease in useful effect—in other words, as the liquor becomes more dilute, the proportion of salts eliminated from the molasses becomes smaller, and the loss of sugar into the osmose water greater. In order to avoid this difficulty the author adopts a novel arrangement which he designates "evaporative osmogene with strong osmose." The upper parts of each of the 100 constituent frames of the osmogene are so arranged as to form a reservoir of liquid above all the frames, and this reservoir communicates directly with the molasses cells of the osmogene, and is supplied with a coil of closed steam pipes. By this means evaporation is maintained in the liquor undergoing osmosis, and the dilution which would otherwise ensue is counterbalanced. Full description with diagrams is given.—A. J. K.

Improvements in Osmose Apparatus. C. D. Abel, London. From H. Leplay, Paris. Eng. Pat. 9243, June 20, 1884.

This invention relates to an improved construction of apparatus for the osmose treatment of saccharine and other liquors, which is termed "steam osmogene with triple action," and which produces simultaneously the three following results: (1.) The separation of the salts contained in the liquors treated. (2.) The evaporation of the liquids of exosmose. (3.) The evaporation of the osmose liquids. The simplest form of this apparatus is as follows:—A metal tank is divided by a horizontal perforated metal partition, on which is placed the parchment paper, into an upper and lower compartment; the latter serves to evaporate the liquid of exosmose, and to furnish the steam required for the osmose action. The upper compartment receives the liquid to be subjected to the osmose treatment. The lower compartment is supplied with a steam coil, supply and discharge pipes, cocks, etc. The action of the apparatus is as follows:—The lower compartment is in the first instance charged with water to sufficient depth to cover the steam coil, whilst into the upper compartment is introduced a layer of molasses or other liquor to be osmosed. The apparatus can be worked either continuously or intermittently; in the latter case the water in the lower compartment is heated to boiling point by the steam coil; the steam produced is condensed against the underside of the parchment paper diaphragm, and is partly absorbed by the current of endosmose passing to the molasses through the diaphragm, while the other portion mixes with the salts of the exosmose current, and falls back into the lower compartment. The molasses become heated, and increase in volume and decrease in density in consequence of the absorption of the water by the current, and rapidly reach 30° B. Arrived at this point, a kind of equilibrium ensues. About the same quantity of water which the molasses receive is separated by evaporation from the surface. As the operation proceeds, a large quantity of the salts passes from the molasses into the exosmose water in the lower chamber; when the required purity is obtained, the osmosed molasses is run off, and a fresh charge put in, whilst the same water in the lower compartment can be used over again until it is thoroughly charged with salts. When operating continuously, a small continuous stream of molasses is admitted into one end of the upper compartment, and escapes at the other end. As the condensation of the steam in the lower compartment will not be effected if the temperature of the osmosed molasses rises too high, such temperature may be reduced by means of air currents on the surface, or by a zigzag coil of cold water-pipes through the molasses. The osmose apparatus above-described requires to be placed horizontally, necessitating a large amount of surface in the factories, which is often inconvenient. In addition to this, there is often considerable loss of heat, and waste of fuel by the evaporation taking place from the surface of the molasses. To obviate these disadvantages, a second osmose compartment may be placed above the first, and a third again above that, so that the liquor in each cell acts as exosmose to the one above it, and the steam from the lower is in each case condensed on the surface of the diaphragm above it. Another arrangement for still further economising space is as follows:—The exosmose compartment at the bottom is formed, as before, of a horizontal tank heated by steam coil or otherwise. Above this, however, instead of the horizontal perforated plate and diaphragm, there is arranged a series of vertical wedge-shaped pockets (apex down), whose sides are made of wire gauze, lined with parchment paper. At the top, these pockets unite to form one large shallow vessel, which, as before, contains the molasses or other liquor to be osmosed. By this means much larger surface is obtained, but otherwise the process is conducted as described in connection with the more simply-constructed apparatus.—A. J. K.

Improvements in Means and Apparatus for Bleaching Sugar. C. H. Day, High Holborn. From C. O. Foster, Brookline, U.S.A. Eng. Pat. 1972, February 12, 1885.

The invention consists of a special apparatus for the treatment of crystalline or granular sugar by sulphurous acid gas. The sugar is made to pass slowly through a rotating drum, provided with shelves inside for regulating the motion of the sugar. The sulphurous acid gas is generated by burning sulphur in a small furnace, and after being cooled by suitable means, is forced into the cylinder with the sugar by means of a jet of dry steam or of air. At the other end of the cylinder the sugar is delivered into a hopper, and the gas escapes up a flue.
—A. J. K.

Improvements relating to the Preparation of Food for Animals from the Refuse of Breweries, or of Starch and Glucose Factories, and Apparatus therefor. W. R. Lake, London. From F. W. Weisebrock, New York, U.S.A. Eng. Pat. 2208, February 17, 1885.

BREWERS' grain, as well as the refuse from starch and glucose factories, are well known to be wholesome food for cattle, provided they can be used in a fresh state. These bodies contain carbohydrates and albuminates, and on keeping for any length of time, are liable to undergo fermentation, with formation of lactic and other acids. The author states that the various processes which have been devised for drying brewers' grain and similar materials have failed to produce a sweet and wholesome food, owing to the fact that the air used for drying has not been freed from active fermentation germs. As a result the article contains a large proportion of dry lactic acid, which is no more desirable than the same material in the moist state. It is claimed that by the invention herein described this objection is overcome. The brewers' grain or refuse is first mixed with salt or salt water in proportion of 5lb. salt to 1 ton of the grains, with the object of keeping the latter sweet during transit from the place of its production to the drying apparatus. The wet grains thus salted are next subjected to the action of dry air, from which all active germs have been previously removed by exposure to a temperature of over 400° F. By this means, when thoroughly dried, a sweet and wholesome food is obtained. Special apparatus, suitable for carrying on the necessary operations, is fully described.—A. J. K.

Improvements in Apparatus or Devices for the Treatment of Sugar Cane. H. J. Chapin, New York, U.S.A. Eng. Pat. 2553, February 25, 1885.

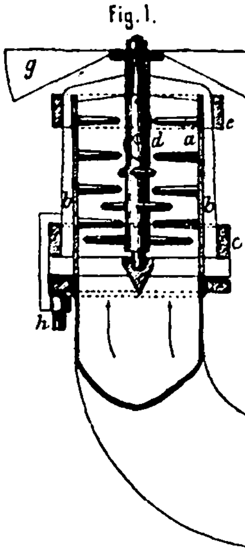
THE inventions described under this specification "consist of a cane shredder of improved construction and mode of arrangement, of improved devices for conveying the shredded cane and juice from the shredder to the juice extractor, and of the more convenient arrangement—relatively to each other—of the mechanical elements or members of the combination therein described." Full description, with copious reference to diagrams, is given of the construction and arrangement of machinery above mentioned.—A. J. K.

XVI.—BREWING, WINES, SPIRITS, Etc.

Apparatus for Spirit Distilleries. Dingl. Polyt. J. 255, 345.

A DISINTEGRATOR, patented by Gaul and Hofmann (Ger. Pat. 27,351, September 19, 1883), to be connected with the delivery pipe of a Henze steaming apparatus, is shown in Figs. 1, 2, 3. The disintegrator is clamped by *h* to the delivery pipe. The steamed grain as it leaves the disintegrator falls upon the wheel *g*, which gives a rotary motion to the graters fixed upon the spindle *d*.

Fig. 1.



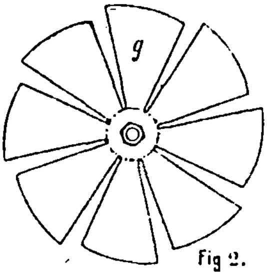


Fig. 2.

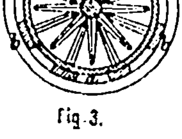


Fig. 3.

The disintegrator patented by Hall (Ger. Pat. 27,807, November 22, 1883), illustrated by Figs. 4 and 5, is intended to be attached at A to the delivery pipe of the steaming apparatus. Supported by *d*, there is a double grating, of which the under half D is stationary, but the upper half E movable in a direction at right angles to the bars by the screw at the end of FG. The slits of the grating can thus be closed entirely, or their width adjusted at will. The side H can be easily removed for the purpose of cleaning the gratings. Upon opening the valve C, the contents of the steamer enter B, and by the steam pressure are forced through the slits of the gratings in a very finely divided condition, stones and large or hard particles being caught by E.

Fig. 4.

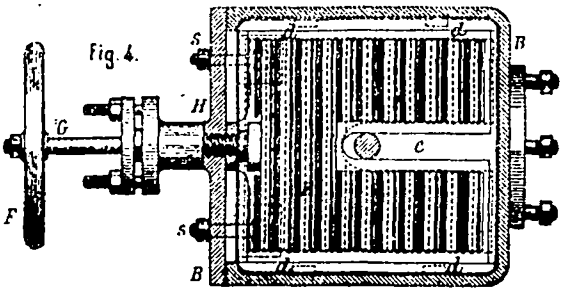


Fig. 5.

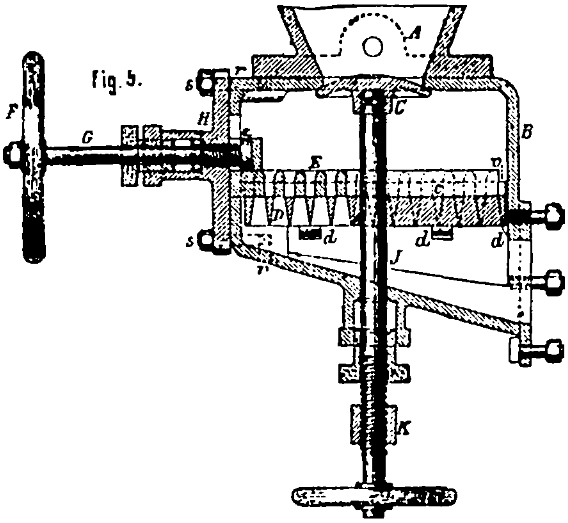
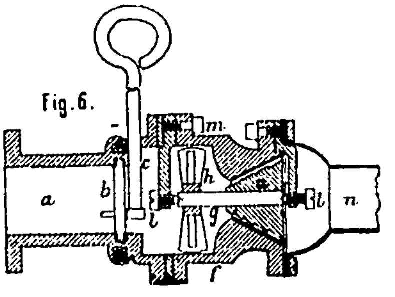


Fig. 6.



Wösler's disintegrator (Ger. Pat. 28,278, December 21, 1883) is shown in Figs. 6 and 7. It is attached at *a* to the delivery tube of Henze's steamer. Stones and similar substances are caught by the movable grating *b*, which is kept clean by the rake *c*. Accumulated stones are removed at the manhole *e*. The ribbed cone *u*, forming the grinding apparatus, is rotated by the wheel *h*, which the rapid flow of material from the steamer sets in revolution. The tap *o* serves for the introduction of water

Fig. 7.

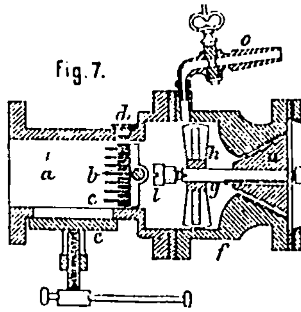


Fig 8.

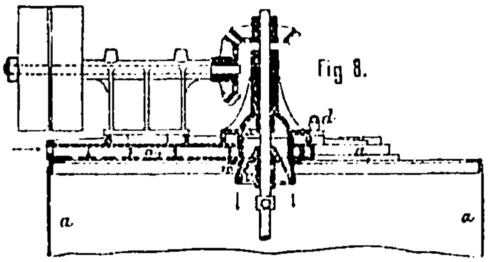


Fig 9.

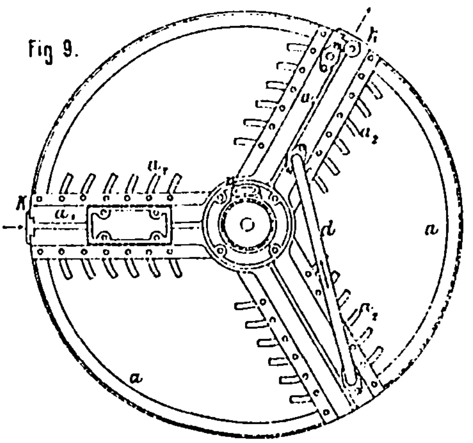
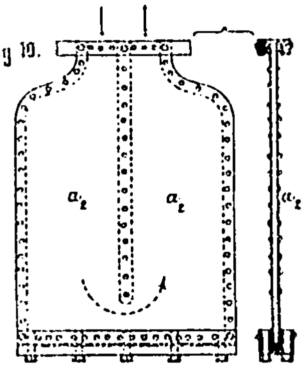


Fig 10.



The washing and cooling apparatus patented by Hampel (Ger. Pat. Addition 28,891, February 5, 1882) is shown in Figs. 8, 9, 10. The cooling water enters at K, and takes the course indicated by the arrows. The steamed grain or other material is forced by steam pressure through the tube *u* into the vessel *a*.

—W. D. B.

Liquorice Root in Beer Brewing. R. Kayser. Dingl. Polyt. J. **255**, 538.

It has been found by experiment that 1 part of liquorice root imparts to water the same degree of sweetness as 8·5 parts of sugar-candy. Thus in sweetening efficiency one part of glycyrrhizin is approximately equal to 140 parts of sugar-candy. Since glycyrrhizin is not affected by fermentation like sugar, the addition of 1 kilo. of liquorice root to 500 litres of wort will give a sweeter beer than if 8·5kilos. of sugar had been used.

—W. D. B.

Analysis of Pure Wine. W. Klinkenberg. Zeits. Anal. Chem. **23**, 514.

Samples of wine from Saar were analysed by the following method :—100cc. were distilled with milk of lime to fix any CO_2 present, and the alcohol estimated from the specific gravity of the distillate. The amount of extract was estimated by evaporating 50cc. to a syrup on the water bath, and drying in a steam oven. The acid present, after freeing from CO_2 by boiling, was determined by titration with baryta solution. For the mineral constituents 100cc. were taken in each case. CaO and MgO were determined directly in the wine, the sulphuric acid after decomposing organic matter with $KClO_3$ and HCl, chlorine, phosphoric acid, and the alkalis in the ash. Wild's instrument was employed for polarisation.—J. B. C.

Estimation of Wine Extract. C. Weigelt Rufach. Zeits. Anal. Chem. **24**, 26-30.

Wine extract is defined as the sum of the non-volatile substances. The author discusses the relation existing between the sp. gr. of the solution and the weight of the extract, and a tabular statement is appended showing that the indirect method for estimating the amount of extract is comparable with the direct method in wines of very various qualities and specific gravities. The percentage of extract can also be calculated by finding the sp. gr. from Balling's saccharimetric tables, and the numbers so obtained agree satisfactorily with those obtained by the other two methods.—S. R.

New Apparatus for the Treatment of Hops. Dingl. Polyt. J. **255**, 165.

A boiler, devised by A. Steinecker, is shown in Figs. 1 and 2. It is furnished with a double bottom ; while the bottom, sides and lid of the hop-basket are made of perforated tin or wire-netting, as in the boilers A and B, or it is, instead, only an enclosed sieve, as in the boiler C. In the centre of the boiler is a tube *l*, provided with a head-piece, which scatters the boiling water around, and by means of which the hops are kept continually soaked during the boiling. The vessel being filled with fresh hops, the cock *c* is opened to admit steam from the pipe D, while the cock *f* is also open to allow of the exit of the steam and volatile oil of hop, which pass by the pipe *g* beneath the surface of the wort. After the steaming, hot water is injected through the cock *o* and the rose *n*. This water, together with dissolved volatile oil and condensation water from the steam, passes into the wort by the cock *e* and tube *g*, the cocks c and *a* now being shut. If the steam and scalded hops are to be heated in the next boil, the steam-cock *a* is opened, water having been already admitted the day before and left to stand. This water is soon raised to the boiling point. While the lid *m* is still closed, the cock *f* remains open, so that the steam produced can reach the wort through the tube *g*. The steam-cock *a* is closed after each boil, *f* remains open, *c* is opened and the aqueous extract is drained off into the wort-pan. The boiling with water having been repeated from two to four times, the lid is removed, and the hops are transferred to the wort. According to A. Kempe, of Moscow, 55kilos. of hops are sufficient for one boil, in place of the 70kilos. generally used by brewers. At the beginning of a boil, 33kilos. of hops are thrown into the wort; this is boiled for three to three and a half hours, and then transferred to the cooler in the usual manner. Twenty-two kilos. of hops are placed in the inner iron- or tinned copper boiler A (Fig. 3), provided with a stirrer D, and either wort or water is now added through the pipe F, the fluid penetrating also into the spaces B and C, through the sieves JJ. When all the valves are closed, steam is allowed to enter by the cock K in the casing A_1. The hops and fluid in A are hereby brought to the boiling temperature, or even higher, the amount of pressure being indicated by the manometer M. In order to quicken the exhaustion of the hops, the valve *o* is opened, so that the fluid passes through the depositing vessel V and the tube *v* into a cooler placed above the boiler, flows through the spirals present therein, then through the gauge-glass S, and the valve O, into the chamber B, and finally, through the sieve J, back into the chamber A. Should sufficient

Fig. 1

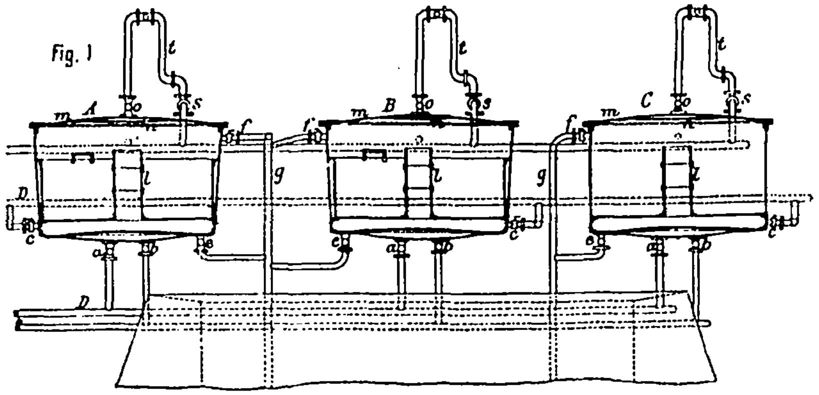


Fig. 2

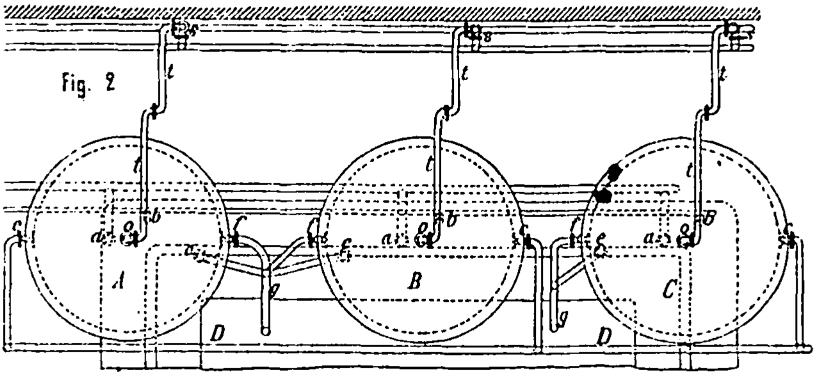
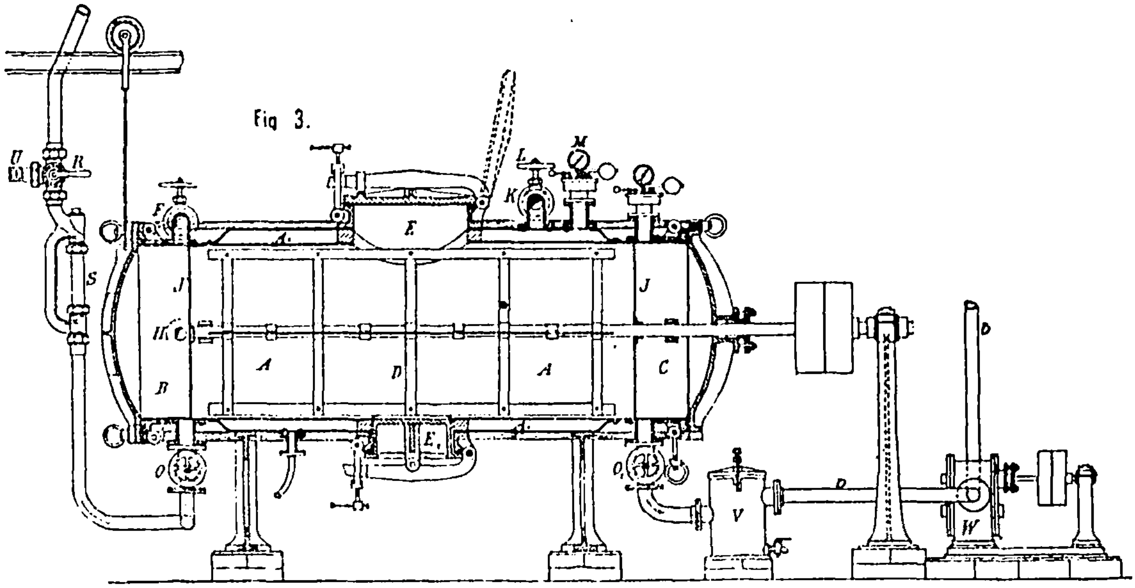


Fig 3.



cold water for the cooling not be at hand, so that this circulation cannot be effected, the pump W is employed. Hünerkopf & Son and F. Rutschmann, of Nüremberg, do not at once add the entire hops to the hot wort, but dry them at a temperature of not more than 38° in the drying apparatus shown in Fig. 5, until they become greatly disintegrated and fall asunder into powder under the action of a rake. The drying chamber is constructed so that the temperature can be regulated with the greatest nicety. In order to effect the removal of moisture from the hops as quickly as possible, passages (*ii*) are constructed beneath the chamber for the admission of cold air. The chamber is further provided with two thermometers *ff* (Fig. 7), and with sliding doors c and c_1, which latter serve to screen off the fire b when necessary. The box g is made of fine perforated tin, and is filled with hops from the trap-door h. When the drying is completed, the slide m is pulled up and the hops are allowed to fall from g on to the cylindrical rake n, and to pass from thence, in a finely divided condition, into sacks ready to receive them.—E. G. C.

Fig 4.

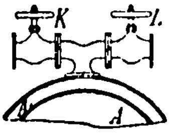
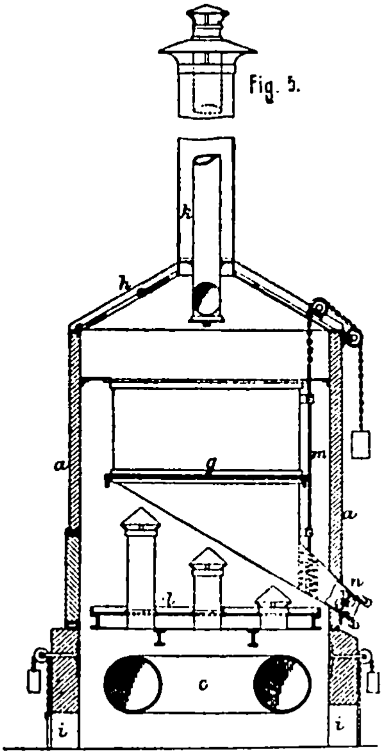


Fig. 5.



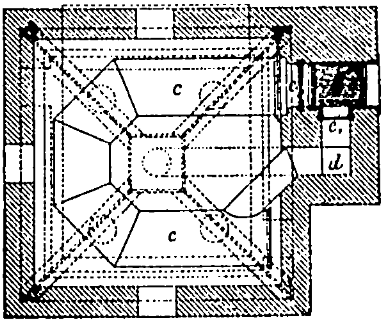
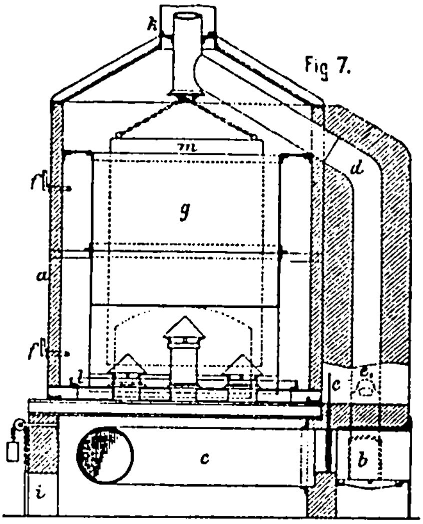


Fig. 6.

Fig 7.



Contributions to our Knowledge of Caramel. C. Amthor. Zeits. Anal. Chem. **24**, 30-33.

Paraldehyde destroys the colour of an alcoholic solution of caramel, and forms at the same time a brown precipitate. This reaction is made use of by the author for determining the amount of caramel added to wines and spirits. A standard solution of caramel is prepared by caramelising 10grm. of pure cane sugar and dissolving the product in water. Any undecomposed sugar is then destroyed by fermentation, and the solution is diluted to 100cc. A solution of paraldehyde in absolute alcohol completely destroyed the colour of this solution after twenty-four hours. With pure white wine, natural wine, and extract of raisins, paraldehyde gives a white precipitate; the caramel precipitate can be further identified by its reaction with phenyl-hydrazine. A solution containing two parts of phenyl-hydrazine hydrochlorate with three of sodium acetate dissolved in twenty of water, gives with solutions of caramel a brown precipitate soluble in warm ammonia, or weak caustic soda to a red liquid, and is reprecipitated from this alkaline solution by the addition of hydrochloric acid. The precipitate is soluble in concentrated hydrochloric acid or nitric acid, but is reprecipitated on diluting with water. Alcohol similarly dissolves it, and water reprecipitates it after some time. The method adopted in testing for caramel in white wines or spirits is as follows:—10cc. of the liquid (if a wine 10-15cc. of alcohol are first added) are placed in a tall glass cylinder, and 30-50cc. of paraldehyde solution added, together with sufficient absolute alcohol to make the two liquids mix. After twenty-four hours the precipitate is collected, washed with alcohol, and dissolved in warm water. The clear filtered solution is then evaporated to 1cc.—the intensity of the colour is a measure of the amount of added caramel. With very small quantities it is best to evaporate the solution over sulphuric acid in the receiver of an air-pump. The solution must not be warmed, or a caramel-like precipitate is obtained even with the purest wines. The concentrated solution of the paraldehyde precipitate can then be treated with phenyl-hydrazine solution, when the caramel phenyl-hydrazine precipitate is thrown down. If the solution is weak it first only becomes cloudy, but the precipitation is complete after standing for a day. Any resinous bodies present can be dissolved out by ether, which has no action on the precipitate. Phenyl-hydrazine, unlike paraldehyde, gives no precipitate with natural wines. Wines containing a large percentage of sugar should be diluted before trying the above reactions.—S. R.

The Sugar and Acids in Must. E. Schmidt. Zeits. Anal. Chem. **24**, 33.

The following table shows the percentage of sugar and acid in the must from Montreux and Villeneuve during the last five years:—

Montreux.	Sugar %	Acid %	Villeneuve.	Sugar %	Acid %
1880	15·2	0·9	......	......	......
1881	17·8	1·1025	......	......	......
,,	17·5	1·0867	1881	18·525	1·1025
1882	16·6	1·085	1882	16·726	0·9075
1883	16·9	1·085	1883	17·64	1·1025
1884	15·7	0·915	1884	15·23	1·005
,,	16·8	0·924	,,	15·	1·0935

Last year's must from Villeneuve is exceptionally low in sugar, since owing to a hailstorm a great part of the grapes had to be plucked before they were quite ripe. —S. R.

Improvements connected with Preserving, Storing, and Aerating Lager and other like Beers. W. Aubert, jun., Balham. Eng. Pat. 5747, March 31, 1884.

The beer is stored in iron drums having a pipe reaching to the bottom, through which carbonic acid under pressure is forced in, so as to charge the contents. The pipe also serves as an outlet when the contents of the drums are discharged. A modification of the apparatus is also described and shown, in which a spherical vessel is placed in the interior of the drum, charged with carbonic acid gas or air under pressure; a two-way cock is so arranged that the gas is used for forcing the liquor from the drum when the outlet cock is opened. The claims are two, for the apparatus as described.—C. C. H.

Making Fodder from Distillers' Wash. Jasper Wetter, London. Eng. Pat. 6768, April 24, 1884.

The wash is submitted to the action of a spray of glue or gelatine solution, which carries down most of the nutritious matter. The precipitate is allowed to settle, the supernatent liquid run off, carbonate of lime is added to the residue nearly to neutralisation, which is completed by sodium bicarbonate, and the neutralised mass is raised by an elevator and caused to flow down over the surface of heated rollers, and then over an endless drying band until sufficiently dry to be ground by crushing rollers. The roller drying apparatus, and the means of heating it, are claimed as well as the process.—J. M. H. M.

Utilisation of Hops in the Manufacture of Intoxicating Beverages otherwise than Beer. Max Hedicke, London. Eng. Pat. 7478, May 9, 1884.

The object of this patent is the preparation of a hop-liquer. The extract may be made by maceration, digestion, distillation, or a combination of any such processes without materially affecting the result. The patentee prefers to proceed as follow:—7lb. of hops are macerated in 60 quarts of spirit 7° over proof, and after twelve hours the extract is removed; the partially spent hops are then distilled with 40 quarts of spirit, and the distillate mixed with the extract first obtained. Brandy or whisky, etc., and sugar and water, are then added according to taste. The process of manufacture as described is claimed.—C. C. H.

A Novel Non-alcoholic Drink. T. F. Wilkins, Chiswick. Eng. Pat. 83, January 2, 1885.

This specification describes the preparation of three different liquors for consumption as beverages composed of infusions or decoctions of kola nut roasted, maté, coca leaves, and hops. Different proportions of the various ingredients are used according to the circumstances of the intended consumption. The claims are: (1) For a beverage composed of the decoctions described, with the addition of a small quantity of salicylic acid; (2) a similar beverage, but with the omission of the decoction of hops.—C. C. H.

XVII.—PRESERVING FOOD, MEATS, Etc.

Arresting or Preventing Fermentation. F. S. Barff, Middlesex. Eng. Pat. 5096, March 18, 1884.

Fruit or vegetables are heated with or without water to a temperature somewhat under the boiling point, and from 2 to 4 per cent. of boroglyceride is added. Two per cent. of boroglyceride added to weak wines prevents the second fermentation, and enables them to bear transportation as well as the stronger wines.—J. M. H. M.

XVIII.—SANITARY CHEMISTRY, DISINFECTANTS.

Apparatus for Use in the Process of Softening and Purifying Water. W. Wyatt. Eng. Pat. 6213, April 10, 1884.

The apparatus is to be employed for the purpose of mixing lime-water or other liquid or liquids for use in the said process, or for mixing such liquids with lime or other solid matters. A hollow standard, capable of revolution on its axis, and provided near its bottom with a hollow perforated arm standing out at right angles, is placed in a tank, preferably near the floor, so that the apparatus may be immersed in the materials to be acted upon. At the top of the standard is an arrangement by means of which a stream of water can be injected under pressure, together with a quantity of air drawn in through apertures. The air and water pass down the hollow standard and along the arm until they reach the perforations, through which they escape, causing a more or less violent agitation of the liquid by which the arm may be surrounded. The apparatus may be placed in the centre of a tank, or two or more of them may be placed in one tank, motion being given to them by suitable contrivances. In place of a stream of water under pressure, air may be forced from a pipe through the hollow standard. —E. G. C.

Improvements in Apparatus for Use in the Treatment of Sewage and in other Operations in which Solid Matter is required to be Added to or Mixed with Fluid Matter in Certain Relative Proportions. S. K. Page, C. E. Robinson, and W. Stevens. Eng. Pat. 7198, May 3, 1884.

According to this invention, the flow of the sewage or other fluid, to which the solid matter is to be added, or with which it is be mixed, is caused to actuate apparatus by which the latter is supplied or by which its supply is controlled. The mixture of the solid and liquid matters is thus effected automatically. A vessel of the required form and size is mounted on pivots or trunnions, in such a position relatively to the mouth of a pipe from which the fluid issues, that the fluid so issuing will pass into the vessel. The latter is counterbalanced in such a manner that, when it has received a certain quantity of fluid, it turns on its pivots or trunnions and discharges the fluid received; it then returns to its original position and is ready to receive another charge of the fluid. Above the vessel is mounted a hopper containing the solid matter to be mixed with the fluid, and provided in its lower part with slides or valves, one above the other, and at such a distance apart that the space enclosed between them will hold the quantity of solid matter required for each charge of the sewage or other fluid. These slides or valves have openings in them to permit of the passage of the matter from the hopper when the openings are brought within the mouth of the latter, and they are so connected with the vessel already described that, when it turns on its pivots to discharge its contents, they slide in one direction, the lower valve closing the mouth of the hopper and the opening in the upper valve being brought within the hopper. On the vessel returning to its original position, the upper valve is closed and the opening in the lower one is brought within the mouth of the hopper. The matter previously enclosed between the two valves thus falls into the mounted vessel. The capacity of the lower part of the hopper between the two slides or valves can be increased or diminished as may be required.—E. G. C.

Preventing the Emission of Metallic Vapours, Compounds of Metals, Sulphur Dioxide, or other Vapours from the Chimneys and Flues of Chemical, Metallurgical, or other Works. E. H. Cook. Eng. Pat. 7731, May 15, 1884.

This invention consists in the use of sulphuretted hydrogen, alkaline sulphides, and mixtures of these for preventing the vapours given off from furnaces leaving the stacks, and thus getting into the atmosphere. The sulphur compounds are produced from alkali waste, and the modes of treating the waste are as follows: 1st, The substance is moistened with water, and air or steam blown through the mass. The gases escaping at the outlet are thus charged with sulphur compounds, and being brought into contact with the gases or vapours given off from furnaces, chemical changes occur, resulting in the deposition of the metals present as sulphides, and also in the decomposition of the sulphur dioxide, sulphur being deposited. 2nd, The waste having been mixed with water, cloths are steeped in it, and these are suspended in the flues, etc., when similar changes will occur as before. 3rd, The waste is mixed with water, and the gases and vapours from furnaces are made to come into contact with the mass.—S. H.

An Improved Preparation of Carbon, Adapted Especially for Electrical and Filtering Purposes. S. J. Coxeter and H. Nehmer. Eng. Pat. May 22, 1884.

Carbon and peroxide of manganese, or any other substance capable of giving off oxygen, is mixed with silicate of potash or soda, so that the compound forms a pasty mass. After drying, it is immersed in a solution of an ammonium salt, or chloride of metals, or in any acid that will have the effect of decomposing the alkaline silicate, so that the silica be precipitated intimately amongst the particles of the carbon, and the other substances employed. The mass is then boiled in water, which dissolves out everything but carbon, peroxide of manganese, and silica. After this it is dried and fit for use, especially for elements in electrical batteries. The inventors term their finished product "Silico-carbon."

—S. H.

Improvements in the Purification of Water. W. Anderson. Eng. Pat. 5496, November 23, 1883 (Amended Specification dated May 23, 1884).

This invention relates to the treatment of water by means of spongy iron. According to the method at present adopted, the purifying material is arranged in layers, as in an ordinary filter bed, the disadvantages of this system being that the spongy iron, when used with bad water, becomes covered with slime and caked into lumps, that it has to be disturbed and cleaned from time to time, and that, "the process of purification by percolation being very slow, large areas of filters are required, and a heavy capital expenditure in the purifying material." The inventor proposes to place the spongy iron in a revolving vessel, mounted upon hollow trunnions, which serve as the inlet and outlet for the water. The vessel referred to is not nearly filled with the spongy iron, the particles of which must be free to be carried up on to shelves or ledges within the vessel, and to fall again through the water, fresh surfaces being in this way constantly exposed. The water operated upon flows continuously through the revolving vessel.

—E. G. C.

Apparatus for Disinfecting Rags and other Fibrous Materials. S. W. Parker and H. Blackman, New York. Eng. Pat. 16,539, December 16, 1884.

According to this invention, rags and other fibrous materials may be disinfected while in the bale or package, and liquid or gaseous disinfectants can be caused to penetrate and permeate the mass of the bale in every direction. The disinfecting chamber in which the operation is to be carried out, is furnished with a series of five or more rotary, tubular, perforated screws, which, by means of suitable gearing connected with them, and also with a car upon which each bale in succession is brought into the chamber for treatment, are caused to penetrate the bale to the distance of their full lengths. The hollow interiors of these perforated screws are connected with a separate chamber or receptacle, into which is forced in any desired way a disinfectant, either in the form of gas or liquid. The disinfectant employed passes through the hollow screws and perforations therein, and permeates the whole mass of the bale of rags, etc. When the bale has been subjected for a sufficiently long time to the action of the disinfectant, a stream of pure air can be forced through the screws to drive all noxious gases from the interior of the bale and surrounding chamber. Immediately above the disinfecting chamber is a tank containing a disinfecting liquid, through which all gases and vapours must pass before escaping into the air. Each bale, after having undergone the purifying process, can be withdrawn from the chamber by shifting the driving belts, and so reversing the direction of the rotation of the driving gear connected with the tubular screws and car already referred to.—E. G. C.

XIX.—PAPER, PASTEBOARD, Etc.

Improvements in Bleaching Paper Pulp. J. H. Johnson, Middlesex. From Jean Baptiste Fessy, France. Eng. Pat. 7511, May 9, 1884.

Hitherto the bleaching of materials for paper-making, and the bleaching of other materials, has been effected by immersion in baths of different kinds, whereby there results a considerable loss of the bleaching agents, owing to the large quantities required for immersing the materials. This invention has for its object to effect economy in the cost of bleaching by reason of the small quantities of bleaching agent required, and also to save time, "owing to the rapidity with which the process can be performed by applying the decolourising or bleaching agents in a nascent condition."

The bleaching is to be effected by acting on the materials to be bleached with the solution of the bleaching agent in the form of spray, which may be produced by the action of steam or compressed air. It is proposed to employ for this purpose an apparatus similar to "Körting's Pulveriser," but constructed as described in the drawings accompanying the specification, which should be consulted for details.

"This system of bleaching can be readily applied by utilising the apparatus ordinarily employed in paper-mills, and requires no special skill or training on the part of the workmen. The effect produced on the materials is permanent; and paper pulp, usually very difficult to bleach, can be bleached with rapidity, the operation lasting from a few minutes to a few hours, according to the nature of the materials. Pulp which, owing to the chemical nature of the colour or the mechanical condition of the materials (*e.g.*, wood pulp), hitherto could not be bleached in a practically available manner, can be successfully treated by means of this process; and facility is also given for obtaining an absolutely regular and uniform bleaching, to any desired tint or degree, as the chemical and mechanical action can be arrested at any desired stage." Inventor may use as a means of pulverising any other chemical or mechanical agent; and any bleaching agent may be employed. A saving of 60% in bleaching powder is claimed by the patentee.

—H. A. R.

Improvements in the Manufacture and Subsequent Treatment of Sensitive Paper for Copying Drawings, Documents written on one Side, and the like, by Photography H. J. Shawcross. Eng. Pat. 8771, 1884.

This invention has for its object the obtaining of exact copies of line drawings or photographs on sufficiently translucent paper in black lines, or in some cases of coloured lines, on a white surface, and consists in spreading on the surface of the paper two chemicals, one of them very sensitive to light, and both capable, when combined, of colouring the paper. The inventor uses, for the sensitive solution, a mixture of per-chloride of iron, gelatine, ferric sulphate, sodium chloride and tartaric acid; for the developing material, powdered tannic acid. The surface of the paper is first covered with the sensitive solution, thoroughly dried, and the developing material is then rubbed or pressed in. In using paper prepared in this way, the tracing must be laid over the sensitive surface and exposed to light in a glass frame. After some time the paper is taken out, laid on the surface of water, wetted on both sides, well washed and dried, when it will contain an exact copy of the original tracing. If a deep blue be required, ferrocyanide of potash can be used instead of tannic acid; if a deep red be required potassium thiocyanate may take its place; catechu will give a green.—S. H.

Improvements in Treating Vegetable Substances in order to obtain Fibrous Materials for Textile Purposes, and Pulp for Making Paper, and for other Useful Applications. Thomas Graham Young and John Pettigrew, jun. Eng. Pat. 14,735, November 8, 1884.

THE process consists in treating wood or other vegetable substances with a 20 per cent. solution of nitrous acid, preferably at a boiling temperature. The disintegrated wood is then washed and treated with a solution of caustic soda or other alkali or alkaline earth, with or without pressure. The pulp, after washing, can then be bleached in the ordinary way. The oxides of nitrogen produced in the first part of the process can be recovered by leading them into a tower, where they are acted upon by air and steam.—E. J. B.

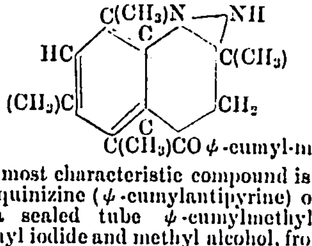
Improvements in Treating Vegetable Substances in order to obtain Pulp for Making Paper, and for other Useful Applications. Thomas Graham Young and John Pettigrew, jun. Eng. Pat. 14,988, November 14, 1884.

THIS process is similar to the above, nitric acid or nitric acid containing nitrous acid being substituted for the nitrous acid.—E. J. B.

XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES AND EXTRACTS.

On Derivates of ψ-cumylquinizine. S. Haller. Ber. **18**, 706.

THE derivatives of cumylquinizine can be prepared in the same manner as Knorr's quinizines (*Ber.* xvi. 2597, and xvii. 546 and 2032), with which they also agree as regards reactions and properties. The constitution of the compounds belonging to this group can be represented, thus:—



ψ-cumyl-methyl-oxyquinizine.

The most characteristic compound is ψ-cumyldimethyl-Oxyquinizine (ψ-cumylantipyrine) obtained by heating in a sealed tube ψ-cumylmethyloxyquinizine with methyl iodide and methyl alcohol, from four to five hours, at 100° C. Its reactions are very peculiar. Its aqueous solution is coloured red with iron perchloride. Nitrous acid produces a bluish green colouration, owing to the formation of isonitroso-ψ-cumylantipyrine. It dissolves with a deep red colour, in cold concentrated nitric acid, which contains no nitrous acid. On adding water the nitro-derivative is separated as a pink crystalline mass.—S. H.

Brucine. O. de Coninck. Compt. Rend. **99**, 107.

ON fractionating the distillate obtained by heating cinchonine or brucine with potash, tetrahydroquinoline is formed. It is an oil of high refractive power, and possesses the smell of quinoline and indol. Tetrahydroquinoline is, therefore, present in the molecule, both of cinchonine and brucine.—J. B. C.

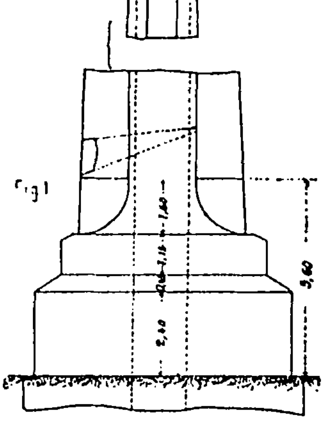
Improvements in the Manufacture of Salicylic Acid. W. Lloyd Wise. From Chemische Fabrik vormals. Hofmann and Schoetensack, Ludwigshafen. Eng. Pat. 3723, November 19, 1884.

CHLORO-CARBONIC acid (carbonyl chloride or phosgene gas) is passed into a dry-heated powdered mixture of 30gs. phenol and 40gs. caustic soda, which is agitated mechanically, and heated gradually from 140° C. to 200° C. The operation is continued till 90 per cent. of the phenol has been converted. The residue is dissolved in water, heated with acid, the phenol driven off by steam, and the salicylic acid precipitated from the sodium salicylate in the usual way. The claim is for causing chloro-carbonic acid (carbonyl chloride) to act as above stated upon dry powdered heated mixture of phenol and an alkali.

XXI.—EXPLOSIVES, MATCHES, Etc.

The Removal of Erections by means of Dynamite. Dingl. Polyt. J. **254**, 456.

THE plan adopted and found successful at Aszod for the removal of a chimney shaft by means of dynamite is described in *Mittheilungen über Gegenstände des Artillerie- und Geniewesens*, 1884, p. 215. In Fig. 1 the details of construction of the shaft are shown; the height was 59·6m. At a height of 6m. above the ground a wedge-shaped hole, 1m. high at its widest end, 0·20m. at its small end, was blasted out, and the chimney fell towards the side in which this cavity was made. In Figs. 2 and 3 the position of the bore-holes is shown, and the following table gives the amount of dynamite No. 2



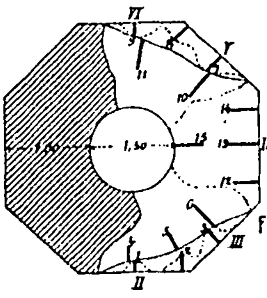


Fig. 2.

used in each hole. The charges were exploded in series, I., II., III., IV. :—

I.	No. 1.	0·20m. deep ...	0·04k. charge.
	2.	0·25 ,, ...	0·07 ,,
	3.	0·30 ,, ...	0·11 ,,
II.	4.	0·25 ,, ...	0·06 ,,
	5.	0·30 ,, ...	0·11 ,,
	6.	0·50 ,, ...	0·50 ,,
III.	7.	0·40 ,, ...	0·25 ,,
	8.	0·30 ,, ...	0·11 ,,
	9.	0·25 ,, ...	0·06 ,,
	10.	0·55 ,, ...	0·50 ,,
	11.	0·55 ,, ...	0·40 ,,
IV.	12.	0·50 ,, ...	0·50 ,,
	13.	0·60 ,, ...	0·80 ,,
	14.	0·50 ,, ...	0·50 ,,
	15.	0·53 ,, ...	0·60 ,,

The charges in the holes 6, 10, 11 were made sufficiently heavy to cut through the brickwork; the shots of group IV. *effected the final displacement, being fired* simultaneously. The work occupied in all three days, and required 4·61k. of dynamite, 15 detonators, 10·2m. of Bickford and 8m. lead fuse. The total cost was 70 marks. Of the 250,000 bricks in the shaft, 150,000 were quite serviceable, and other 20,000 still useful.—W. D. B.

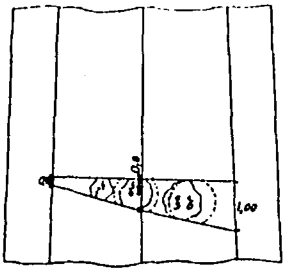


Fig 3

Improvements in Match-making Machinery. Dingl. Polyt. J. **255**, 239.

A MACHINE to obviate the necessity for hand-labour in the dipping or heading of matches has been patented by Holmström (Ger. Pat. No. 25,788, June 30, 1883). To obtain good heads it is not sufficient simply to dip the ends of the splints into the melted composition, but the splints require to be moved about. In the machine patented the arrangement is such that the splints, in the usual carrying frame suspended on an endless band, are led over and pressed by means of a spring upon three or more drums or cylinders dipping into the composition, which, by the revolution of the cylinders, is brought to the ends of the splints. The heads are obtained round and uniform by placing the cylinders at an angle with the line of motion of the carrying frame; the composition is thus pressed against two adjacent sides of the quadratic splint by every drum. A machine for filling flat splints into carrying frames for dipping has been patented by Sebold (Ger. Pat. No. 26,937, July 11, 1883), and is a modification of that devised by the same for the purpose of filling the frames with oval, round, and quadratic splints. The box K (Figs. 5 and 6) is divided into a number of compartments, and rests upon the tube-box K_1, upon the top O of which the splints rest. This top O has perforations corresponding in position with the tubes of K_1, and shaped as shown in Fig. 3, the third form being found most advantageous. The greater diameter of these openings is parallel to the laths L. The tubes of K_1 are filled by shaking the box K, the perforated slide S is brought into such position that the splints from K_1 fall through the perforations, then through the grid R with

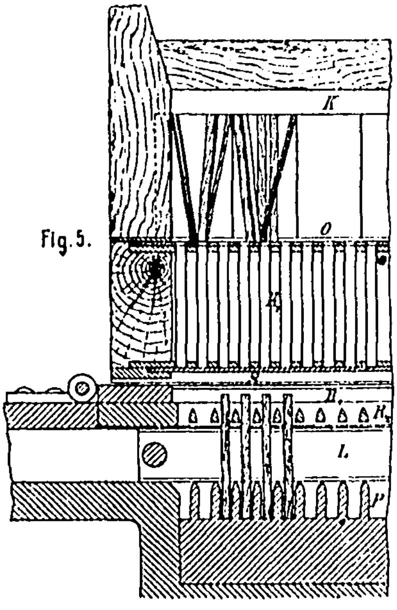


Fig. 5.

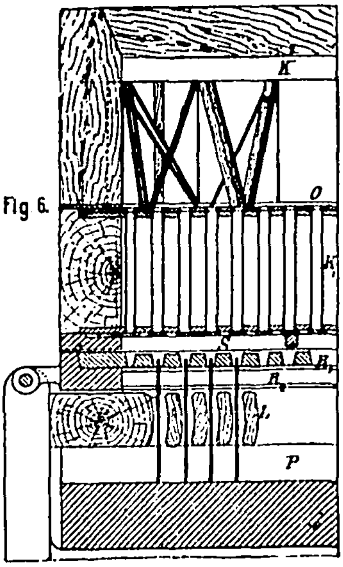


Fig 6.

Fig 3

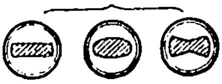
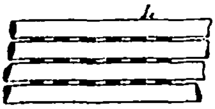


Fig 4



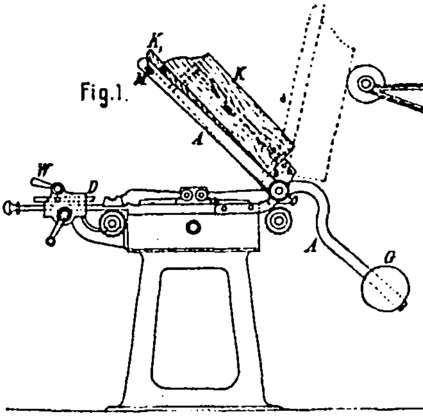


Fig. 1.

narrow openings parallel with L, pass through a second grid R_1, the openings of which are at right angles with those of R, and by this guiding arrangement can be easily and correctly placed between the laths of the carrier, their ends resting in the grooves of P. If the boxes K and K_1, together with the slide S^1 and the grid R_1, be lifted up, the carrying frame may be tightened up in the usual manner and removed from the machine. The surface of the carrier has the appearance shown in Fig. 4. To facilitate the opening and closing of K and K_1, and the shaking of K, there are two levers (Figs. 1 and 2) on either side of the machine, turning around a fixed axis O, provided with counterweights G and connected at the other end by M. The boxes also turn round O, and resting upon M, are moved by the levers. By means of a bolt D fixed in position by the bent lever W, the cross-bar M can be held down, and the boxes K and K_1 being rendered independent of the weights G, can then be readily shaken.—W. D. B.

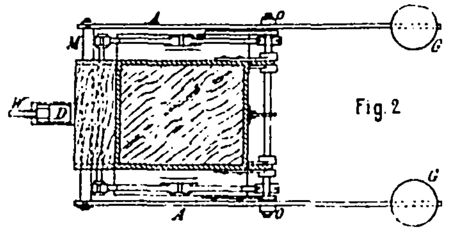
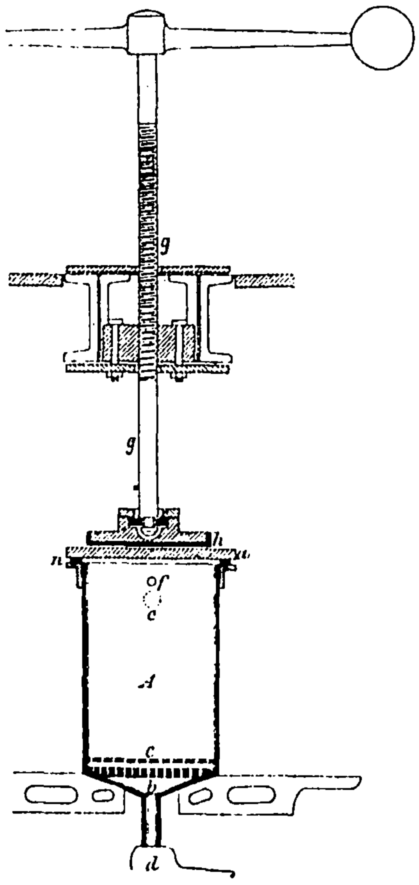


Fig. 2

Improvements in the Manufacture and Application of Explosive Substances. Dingl. Polyt. J. 255, 518.

A DETAILED description, with illustrations, is here given of Lauer's improvements in his apparatus for submarine blasting with unconfined charges (Ger. Pat. 30,242, May 6, 1884; see also *Journ. Soc. Chem. Ind.* iii. [3], 193).



Ballabene prepares a plastic dynamite named lignite by absorbing from 80 to 85 parts of nitroglycerin with from 20 to 15 parts of a nitrocellulose obtained by the nitration of wood-flour. The apparatus shown in the figure is employed for the nitration of the wood. The vessel A is lined with lead, and has a false bottom consisting of two perforated plates, *c* and *b*, between which there is a layer of gun-cotton. The nitration acids, consisting of 62 parts of nitric and 142 parts of sulphuric acid, are introduced into A, *d* being closed, the wood-flour immersed therein, the lid *a* screwed down by *g* to form by aid of the washer an air-tight covering, and after a short space of time *d* opened and about 50 per cent. of the mixed acids expelled by the introduction of air at 2 atmospheres' pressure at *f*. After a short digestion the acids are squeezed out as far as possible by the press *g h*, and the nitrated pulp washed with water. It is to be noted that no arrangement for cooling is applied.

The Societé Anonyme des Poudres et Dynamites proceed according to Lanfrey and Renard's patent in the manufacture of a dynamite with active absorbent, named Paléine. Five qualities, A to E, are issued, of which A contains 40 to 50 per cent. of nitroglycerine, and 60 to 50 per cent. of nitrated straw. A is said to be about ⅕, B about ¼, and C slightly more powerful than ordinary 75 per cent. Kieselguhr dynamite.

Dollink contributes his observations upon the properties of straw nitrocellulose (nitrated straw). Rye straw boiled 15 hours in a 1·2 per cent. solution of soda was nitrated and washed. The product matted together, possessed a brownish yellow colour, contained 11·07 per cent. of nitrogen, and when gradually heated flashed at a temperature of 177°. Exposed to a current of dry air at 60°, signs of decomposition were evident in three minutes.

Guttmann contributes a report upon the explosives manufactured in the Austro-Hungarian Empire. The following is a list of all the blasting compounds at present manufactured, and with the exception of No. 11 the composition of each is given in the original paper:—(1) Black gunpowder, (2) diorrexin, (3) haloxylin, (4) petralite, (5) janite, (6) carboazotine, (7) azotine, (8) amidogène, (9) lederite, (10) vulcanite, (11) myline. In the second series are (1) Nobel's new dynamite, Nos. 1 to 3; (2) white dynamite, Nos. 1 and 2; (3) rhexite, Nos. 1 to 5; (4) Arlberg dynamite, Nos. 1 to 3.

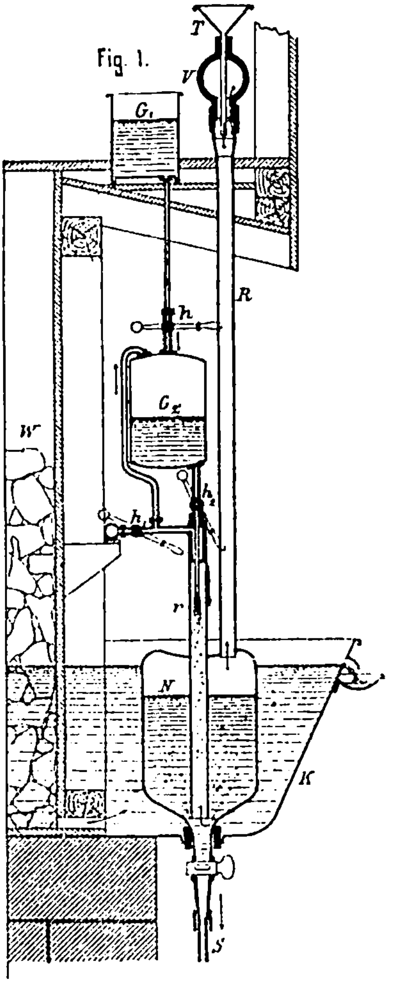
Several instances of the spontaneous inflammation of sponges employed for the cleansing of vessels which had contained nitroglycerin, have recently occurred in several factories where it was the practice to wash the sponges after use in tepid water, then in dilute soda, and dry. It is recommended to lay the sponges immediately after use in water, cleansing them ultimately by soaking in dilute hot soda solution, wringing out several times, and only, after this, to permit them to dry.—W. D. B.

The Manufacture and Application of Explosive Substances. Dingl. Polyt. J. 255, 337.

NORDENFELT and Meurling (Ger. Pat. No. 30,676, Aug. 21, 1884) prepare a carbonaceous substance by subjecting cotton or wood cellulose, loosely packed in a vessel, to the action of a stream of hydrochloric acid gas. The cellulose soon becomes friable, and when the process has been carried sufficiently far, the hydrochloric acid gas is expelled by passing a stream of air. The carbonaceous substance obtained is impregnated with the requisite amounts of sulphur and potassium nitrate, by treatment with solutions of these substances respectively in carbon bisulphide and water; the powder mixture obtained by the evaporation of these liquids is manipulated in the usual manner.

Nitro-compounds for explosive purposes, one in the solid, the other in the fluid condition, are prepared by Gilles (Ger. Pat. No. 27,969, April 14, 1883) by the nitration of molasses. To obtain the solid compound, the molasses, without further preparation, are mixed with the nitrating mixture, the whole thrown into water, and the precipitate washed first with cold then with warm water. To obtain the liquid compound, the patentee advises the nitration of moist molasses of such ultimate composition, that the ratio of carbon to oxygen—taking the oxygen of the water into account—shall approximate 2 : 3. According to Gilles, the liquid nitro-compound is yellow, boils when heated gradually between 180° and 200°, and detonates between 220° and 250°.

Fig. 1.



An apparatus for the preparation of nitroglycerine, patented by Schroeder (Ger. Pat. No. 29,130, December 28, 1883), presents few novel features. An arrangement, by which the glycerine sprayed by means of compressed air was introduced at the bottom of the nitration vessel, was described by Kurtz in 1879.

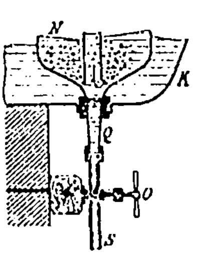


Fig 2.

The following independent analyses of samples, taken from the same bulk of a consignment of Himly's powder, point to the difficulty of properly incorporating the coal-tar pitch with the requisite oxidising salts. One hundred parts of the powder gave to three analysts widely differing results:—

	1.	2.	3.
Potassium chlorate	45·50	33·50	62·50
Potassium nitrate	34·38	48·78	25·00
Coal-tar pitch	20·12	17·72	12·50

—W. D. B.

XXII.—GENERAL ANALYTICAL CHEMISTRY.

Separation and Estimation of Methyl- in presence of Ethyl-alcohol. C. de Poncy. Dingl. Polyt. J. **254**, 500.

The method is based on the different solubilities of the oxalic ethers prepared from oxalic acid in presence of HCl—methyloxalate being easily soluble, ethyloxalate difficultly soluble in water; further, on the insolubility of the amide in water, formed by the action of ammonia upon the ethers.—J. B. C.

Quantitative Determination of Phosphoric Acid in Superphosphates. C. Mohr. Zeits. Anal. Chem. **23**, 487.

The author finds that the method for directly determining the amount of soluble phosphate in commercial "supers" by the ammonium citrate method does not give exact results if the citrate solution is precipitated at once by magnesia mixture, a considerable quantity of phosphoric acid remaining in solution.

The new method is as follows: 5grm. of the superphosphate, or artificial manure, is treated in a mortar with hot water, and filtered into a ¼-litre flask. After extracting three times in this way the insoluble portion is collected in a flask, and digested with 25cc. of an alkaline solution of ammonium citrate for one hour at 60-70°. The alkaline solution is filtered into the first portion, washed, and filled to the mark. If not acid, it should be previously acidified slightly with nitric acid.

To 10 to 20cc. of this solution an equal volume of fuming nitric acid is added, and a large excess of molybdate solution. The whole is digested an hour at 85° on the sand-bath. After cooling, it is filtered, the yellow residue dissolved in ammonia, again filtered, and precipitated with magnesia mixture. The precipitate is either weighed or titrated with uranium solution. The results of analyses by this method agree well with those calculated by difference—*i.e.*, by subtracting the phosphoric left undissolved in ammonium citrate from the total phosphoric acid in the sample—whereas the numbers obtained by the old direct method are in every case about two per cent. too low.

Another advantage of this method is that it can be employed in the case of all manures in which magnesia is present.—J. B. C.

Precipitation of Manganese with Bromine. C. Holthof. Zeits. Anal. Chem. **23**, 491.

The author's method of precipitating manganese with bromine in presence of ammonia and ammonium chloride is a modification of that of Wolff (*Zeitschr.* 22, 520). It is as follows: If iron be present, it is first precipitated with ammonium carbonate, and the filtrate acidified. The solution is then diluted, so that about 1dgrm. of Mn is contained in ¼ litre of liquid. Bromine is gradually added in the cold, until the solution is distinctly red. A large excess of *concentrated* ammonia is added, when the peroxide at once falls down. The solution is heated and filtered. The filtration is done by the pump, and the damp filter transferred to a platinum crucible, dried, and ignited. If only Fe is present the operation is a simple one. With small quantities of CuO, CaO, and MgO, the method gives good results. BaO, CuO, and ZnO may be previously eliminated. In presence of the alkaline earths, peroxide of manganese carries down a quantity of these oxides, as also of the alkalis, CuO, ZnO, and MgO. This error may be partly avoided by re-dissolving the precipitate in HCl, and precipitating with $(NH_4)_2CO_3$; but the separation is not complete, especially when large quantities of CaO are present.

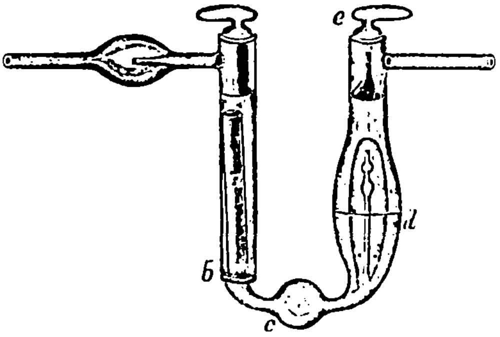
When, however, NH_4Cl is added, and after precipitation, the solution heated, these oxides, if present, are re-dissolved as double chlorides. ZnO and BaO are best separated first. CuO may be determined in the filtrate from MnO_2. If relatively large quantities of CaO and MgO are present, it is necessary to re-dissolve the first precipitate in dilute nitric acid, with a slight excess of formic or oxalic acid to prevent reduction. The excess of organic acid is then destroyed by the addition of a crystal of $KClO_3$, and the solution concentrated. By a further addition of $KClO_3$ the manganese is completely precipitated as MnO_2.—J. B. C.

Fusion Reagent for Silicates. C. Holthof. Zeits. Anal. Chem. **23**, 498.

THE author recommends sodium bicarbonate, 12-15 parts of which are used with the finely-pulverised silicate, a portion being placed at the bottom of the crucible and another portion mixed with the silicate. The crucible is then heated below fusion heat for a short time, and finally the mass is allowed to fuse quietly.—J. B. C.

Substitute for Calcium Chloride Tube. S. Schmitz. Zeits. Anal. Chem. **23**, 515.

THE construction of the apparatus is shown in the diagram. At *b* a piece of platinum gauze is placed, upon which rests a stick of glacial phosphoric acid. The limb *d* contains strong sulphuric acid, and is furnished with an arrangement similar to that of Schrötter's desiccator (the sulphuric acid is filled to a mark previously determined, so that the acid cannot run back). The limbs of the tube are closed by hollow ground stoppers, smeared with a solution of gutta-percha in heavy petroleum. The stopper at *e* is furnished with a conical tube *g*, which prevents bubbles of sulphuric acid being carried away. The bulb *c* retains any liquefied phosphoric acid.—J. B. C.

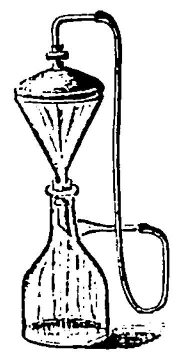


Titrations with Potassium Permanganate Solution. W. Lenz. Zeits. Anal. Chem. **24**, 34-41.

PERMANGANATE solution, when used for estimating organic substances in water, is untrustworthy, since only those bodies which are very easily oxidised are completely burnt up to carbonic acid. With grape sugar, great discrepancies were noticed; the greater the excess of permanganate, the greater the amount used. In no case, however, was the theoretical quantity of permanganate reduced, although about five times the calculated quantity was added. The experiments were repeated by varying the amount of grape sugar added to a constant quantity of permanganate solution, and then boiling for ten minutes, but with similar discordant results. With glycerine the experiments yielded results still further from the truth. The grape sugar never used more than 54 per cent. of the theoretical quantity of permanganate, and the glycerol (glycerine) never more than 34 per cent. under like conditions. The permanganate process is, therefore, useless for either absolute or relative estimations of organic matter. With decinormal permanganate and glycerine equally unsatisfactory results were obtained. With oxalic acid and strong alkaline permanganate solution $\frac{1000}{1000}$ normal, containing 40grm. of KHO to the litre, after twenty-four hours before titrating back, 2 out of 9 experiments were near the truth. The best results were those obtained when twice the theoretical quantity of permanganate had been employed. The oxalic acid solutions used for titrating permanganate should be freshly prepared, as the author finds that centinormal acid loses 15 per cent. of its strength in one week, when exposed to diffused daylight. Normal solutions will keep in the dark if stored in full bottles. Centinormal solutions, even in the dark, lose strength after a few weeks. The addition of 10grm. of boracic acid per litre prevents the decomposition of the oxalic acid, and does not impair its value for titrating purposes. It is best, therefore, to keep decinormal acid containing 1 per cent. of boracic acid, and dilute it as required.
—S. R.

Funnel for Filtration in Absence of Air. F. Allihn, Zeits. Anal. Chem. **23**, 517.

THE arrangement of the apparatus is given in the diagram. The funnel has a cylindrical rim 1-2cm. high, covered with a lid provided in the centre with a neck. Into this fits a cork and bent glass tube. The funnel is fitted into a filter flask, which has a side tube. By connecting the tube of the funnel with that of the flask by a piece of indiarubber tubing, the exterior air is excluded. In case a particular gas is required, the funnel is then provided with a double-bored cork. Through one opening the gas is introduced, and it passes out by the other, a connection being made with the filter flask as before.—J. B. C.



Method for Estimating Nitric Acid Volumetrically. A. Longi. Zeits. Anal. Chem. **24**, 23-26.

STANNOUS salts destroy the blue colour produced by diphenylamine in solutions of nitric acid. The stannous solution employed is made by dissolving 40grm. of potassium stannous sulphate (Marignac) in 800cc. of water, then adding an equal volume of dilute sulphuric acid, and a little concentrated hydrochloric acid. The solution is then titrated, and diluted with weak sulphuric acid, till decinormal. The accuracy of the method depends on the amount of sulphuric acid present. From numerous experiments, the author shows that when 3·5 volumes of concentrated sulphuric acid are added to each volume of the solution to be tested before titrating, the method is quite accurate. Large quantities of ferric salts prevent the application of this method. Concentrated solutions of nitrates should be diluted with water before adding the concentrated sulphuric acid, and in all cases the mixture should be cooled before adding the diphenylamine solution.—S. R.

A Thermostat of Simple Construction which can also be used as a Registering Thermometer. E. H. von Baumhauer. Zeits. Anal. Chem. **24**, 42-46.

AN ordinary glass cylinder A is connected with a cork, or, better, fused to a narrower glass tube B open at both ends, but drawn out at the lower extremity. To the upper end a brass knee-piece is cemented. A round hole is bored in this brass piece immediately above the tube B, so as to allow of the passage of a long graduated tube C into it. The tube C fits air-tight by means of a well-greased leather washer, but can be raised or lowered to any desired height. The gas passes through this tube, and is shut off all but from a small hole in its side about 1½cm. from its lower end by the rising of the mercury in the tube B from the expansion of the air in the glass cylinder. The instrument is graduated by first removing the tube C, and immersing it in a bath at the maximum temperature for which it is required, and then whilst hot filling the tube B with sufficient mercury to prevent air being drawn into the air chamber on cooling. The tube C and the brass piece are next connected, and the gas passes in from the upper end of C, and out from the brass piece by the indiarubber tube Z. On gently heating, the mercury rises in the tube B, and nearly shuts off the gas from time to time as the tube C is raised, the division on the scale coinciding with the mark *m* at the different temperatures being noted. Fig. 2 shows how a fine adjustment can be adapted to the instrument in cases where small changes of temperature are important. In most cases, however, the regulation by hand is all that is needed. To make the instrument self-record the maximum temperature, a small plug of indiarubber is placed in the lower end of tube B which, whilst allowing the mercury to rise freely as the temperature increases, prevents its return. The tube C, which is then connected by a pulley with a counterpoise, rises with the mercury, so that the final position of the scale gives the maximum temperature reached.—S. R.

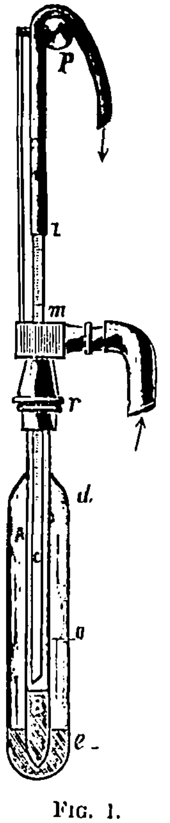


FIG. 1.

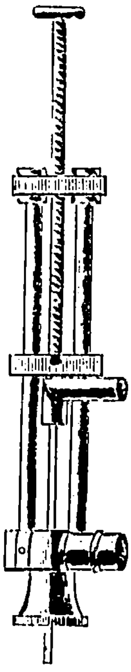


FIG. 2.

Test for Citric Acid. C. Mann. Zeits. Anal. Chem. **24**, 201.

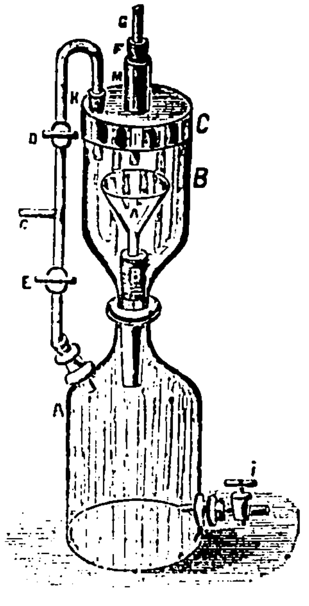
CITRIC acid mixed with glycerol and evaporated to dryness at a low temperature, yields a porous light yellow mass which becomes hard and brittle on cooling; but if treated whilst warm with water, becomes transparent, and has the appearance of swollen-up gelatin. The product is scarcely dissolved by water, ether and alcohol, but readily by alkalis on warming, and is acted upon by concentrated sulphuric acid. The alkaline solution, when concentrated, becomes pale green after some days. If the excess of ammonia be removed from the original solution and hydrogen peroxide be added, the green is produced at once. Fuming nitric acid gives also a green colour, which changes to a blue on warming. When using these reactions as a test for citric acid, the proportion of 1 part of crystallised citric acid to ·6 or ·5 of glycerol is found to be the best. Tartaric and malic acids give no similar reactions.—S. R.

Soap Analysis. A. Gawalovski. Zeits. Anal. Chem. **24**, 219.

IN estimating the fatty acids, the author decomposes with sulphuric acid, transfers to a folded filter, washes and shakes the filtrate and wash-water with light petroleum, separates, and then evaporates the petroleum portion; next he dissolves the fatty acids on the filter with light petroleum, and mixes with the first portion of the fatty acids before finally drying and weighing. The filter need not be dried before dissolving the acids in the light petroleum.—S. R.

Apparatus for Filtering and Drying very Oxidisable Precipitates. W. Bachmeyer. Zeits. Anal. Chem. **24**, 59-61.

THE apparatus is shown in the accompanying figure, and is constructed of glass with the exception of the cover C, which is of brass. The tube *g*, which is connected with the brass tube *m* by the cork *f*, is bent over and unites with a small flask containing the precipitate. This small flask has a cork with three holes, one for the tube *g*, a second for a glass funnel, and the third for a tube connecting with a gas generating apparatus. When all connections are found to be air-tight the tube *c* is connected with a water pump and the cock *e* is opened, the precipitate is drawn over by lowering the tube *g* into the precipitate in the flask as soon as the apparatus is filled with the indifferent gas. The precipitate collects on the funnel *d*, and distilled water can be drawn over to wash the precipitate there, by pouring down the funnel in the small flask. By opening the tap *d* instead of *c*, the filtration can proceed more slowly, and this tap is also useful with very muddy precipitates. To dry the precipitate the cork *k* is replaced by another without an



opening, the glass tube *g* is fused off, and the upper part B of the apparatus removed from the lower part A, and transferred afterwards to a drying oven.—S. R.

A New Separation of Nickel and Cobalt. M. Ilinski and G. von Knorre. Ber. **18**, 699.

If an alcoholic or acetic acid solution of nitroso-β-naphthol be treated with a neutral or hydrochloric solution of cobalt, a bulky purple precipitate is obtained, which resists all action of alkalis, acids, oxidation or reduction. The precipitate is cobalti-nitroso-β-naphthol

$$[C_{10}H_6O(NO)]_2Co.$$

If a solution of nitroso-β-naphthol, in 50% acetic acid, or an aqueous solution of nitroso-β-naphthol sodium, be treated with a solution of a nickel salt, a brownish-yellow precipitate is produced, which is slightly soluble in water and alcohol. Hydrochloric and sulphuric acids deprive it very easily of its metal, leaving behind nitroso-β-naphthol, which remains in solution if sufficient acetic acid is present. If, therefore, an acetic acid solution of nitroso-β-naphthol be treated with little hydrochloric acid, and then with the solution of a nickel salt, no precipitate is produced. On these reactions the following method of separating cobalt from nickel compounds is based:—The solution containing these metals, as sulphates or chlorides, is treated with a few cc. of hydrochloric acid. The liquid is warmed, and a hot solution of nitroso-β-naphthol in 50% acetic acid is added in excess. The precipitate is allowed to settle, and after cooling the supernatant liquid is examined whether it gives a turbidity with a new addition of nitroso-β-naphthol solution. If the precipitation is complete, the precipitate is filtered, after some hours' standing, and washed at first with cold, then with warm 12% hydrochloric acid, until all nickel is removed, and at last with hot water. The filter is allowed to dry, and a few grammes of pure crystallised oxalic acid are added to it. It is then slowly burned in a tared Rose crucible, and the residue ignited in an atmosphere of hydrogen, when pure metallic cobalt is obtained. In the filtrate from the cobalt precipitate, after evaporating the bulk of acetic acid, the nickel can be quantitatively separated by the addition of potassium hydrate. But it is preferable to precipitate in a measured portion of the solution both cobalt and nickel, and, after reducing them in a hydrogen current, to weigh the metals. In another portion, the weight of cobalt is determined by the process as mentioned above, and the amount of nickel can then be estimated by difference. The authors give the results of several analyses, which prove the method very trustworthy indeed. It is also shown that the reaction is far more sensitive than Portman's process with potassium nitrite and acetic acid. Small quantities of cobalt can be determined very accurately when mixed with large quantities of nickel.

—S. H.

A New Method of Estimating Nitrogen, of General Application. Carl Arnold. Ber. **19**, 806.

The author proposes the following method of estimating nitrogen in inorganic and organic substances, with which he has obtained very accurate results with nitrates, nitro-compounds, azo-compounds, amido-compounds, etc. The method is based on the methods which have been already proposed by Tamm-Guyard (combustion with a mixture of soda-lime and sodium-hyposulphite), and by Ruffle (combustion with a mixture of soda-lime, sodium-hyposulphite, carbon and sulphur), but which do not give good results with all substances. It consists in heating the substance with a mixture composed of equal parts of soda-lime, sodium-hyposulphite, and sodium-formiate, the operation being performed like an ordinary soda-lime estimation. The combustion tube is charged with 12 to 15cm. of the mixture and substance, 15 to 20cm. of the mixture alone, and 5 to 10cm. of soda-lime, and heated to a moderate red heat. The operation should last about an hour, and at the termination air is drawn through for five minutes to clear out the last traces of the ammonia. In order to obtain good results, it is essential that the mixture should be packed tightly into the tube (by tapping it in an upright position) and not leave a channel along the top. The proportions of the reducing mixture can be varied considerably, and the sodium hyposulphite can be replaced by 10% of sulphur.

—A. G. G.

On a Colour Reaction of some Diketones. Eug. Bamberger. Ber. **18**, 865.

A few drops of aqueous caustic potash are added to a hot alcoholic solution of the diketone (air being excluded as far as possible); if a dull red colouration is produced, which vanishes on shaking with air, it may be concluded that the diketone used has its two CO groups adjacent. The absence of a colouration, however, is not a positive proof that the CO groups are not adjacent.—A. G. G.

New Books.

On Certain Indications of the Existence of an Allotropic Modification of Elementary Nitrogen, and on the Synthesis of Ammonia. By George Stillingfleet Johnson. London: J. & A. Churchill, 11, New Burlington Street, 1885.

This is an 8vo volume, bound in cloth, containing 32 pages of subject matter. The work is divided into four chapters, and is illustrated by three woodcuts. The chapters are headed as follows:—Chapter I. Historical Electro-chemical Researches. (i.) Direct Synthesis of Ammonia by means of the Silent Discharge. (ii.) Observations of MM. Dehérain and Maquenne, and of MM. P. and A. Thénard, on the Action of the Silent Discharge upon Water Vapour, and upon a Mixture of Nitrogen Gas and Water Vapour. (iii.) Action of the Electric Spark. Chapter II. Researches on the Synthesis of Ammonia from the Gas evolved by the action of Heat upon Solutions of Ammonium Nitrite. Chapter III. Further Experiments on the Nature of the Gas produced by action of Heat upon Solution of Ammonium Nitrite, conducted in 1883. Chapter IV. Experiments with Atmospheric Nitrogen.

The book contains mainly the subject matter of two papers published in the *Journal of the Chemical Society* of 1881, as well as that of three as yet unpublished papers.

Lessons in Elementary Practical Physics. By Balfour Stewart, M.A., LL.D., F.R.S., Professor of Physics, Victoria University, The Owens College, Manchester, and Mr. W. Haldane Gee, Demonstrator and Assistant Lecturer in Physics, The Owens College. Vol. I. General Physical Processes. London: Macmillan & Co., 1885. [All rights reserved.]

This small 8vo volume, bound in red cloth, and comprising besides Preface, Table of Contents, and a List of Tables, 291 pages of subject matter, is the first of three volumes, of which the remaining two, yet to follow, will treat respectively of "Electricity and Magnetism" and "Heat, Light, and Sound."

The work is illustrated by 103 well-executed woodcuts, and the concluding 33 pages of subject matter are usefully devoted to an Appendix, in which the following items receive attention: A. On the Selection, Conduct, and Discussion of operations suitable for the Physical Laboratory. B. Centimetre, Gramme, Second (C.G.S.) System of Units. C. Practical Notes.